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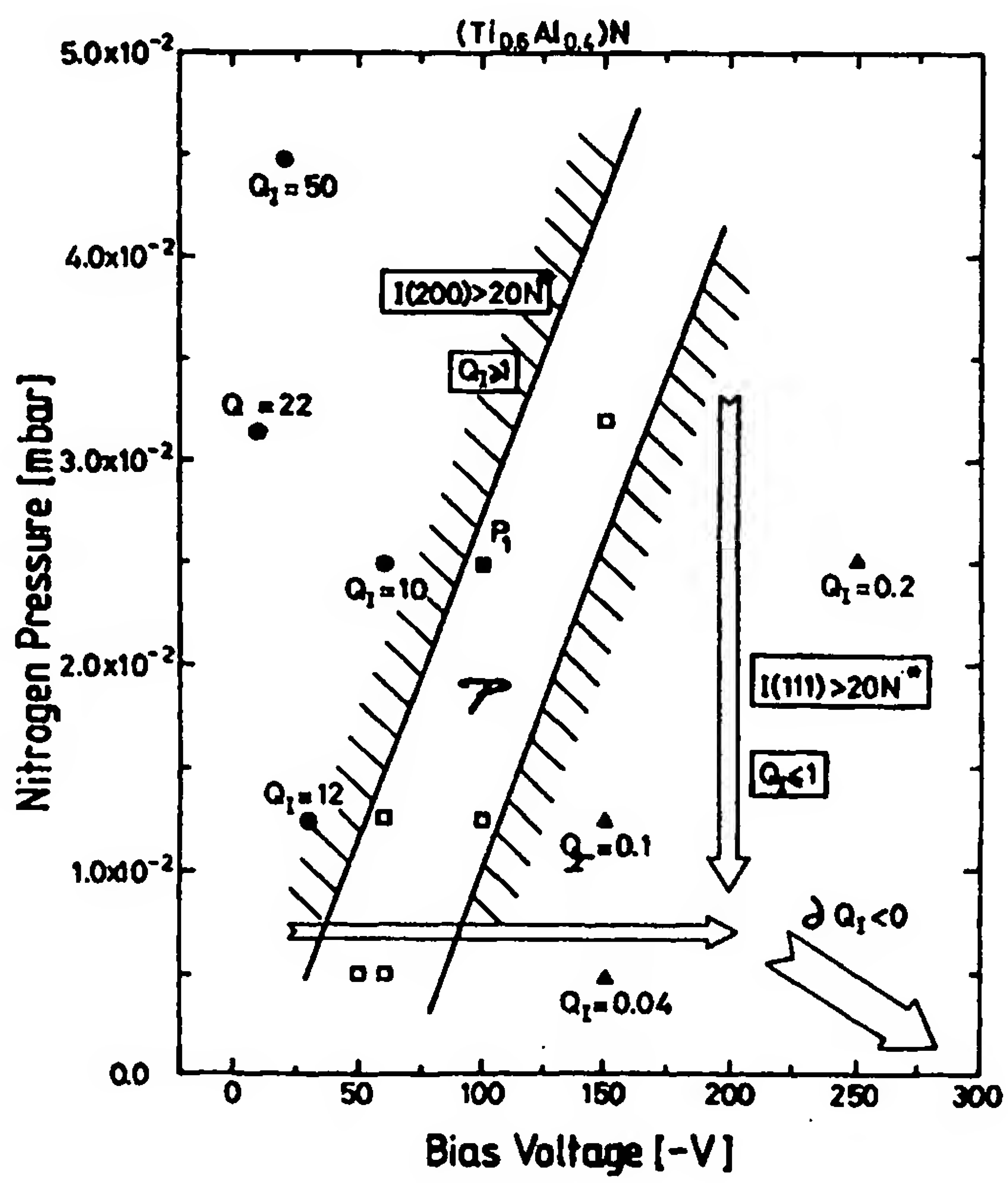
(54) Title: TOOL HAVING A PROTECTIVE LAYER SYSTEM

(57) Abstract

There is proposed a tool with a tool body and a wear resistant layer system, which layer system comprises at least one layer of MeX. Me comprises titanium and aluminum and X is a nitrogen or carbon. The tool is a solid carbide end mill, a solid carbide ball nose mill or a cemented carbide gear cutting tool. Thereby, in the MeX layer the quotient Q_I as defined by the ratio of the diffraction intensity I(200) to I(111) assigned respectively to the (200) and (111) plains in the X ray diffraction of the material using the θ-2θ method is selected to be ≤ 2. Further, the I(111) is at least twenty times larger than the intensity average noise value, both measured with a well-defined equipment and setting thereof.



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TOOL HAVING A PROTECTIVE LAYER SYSTEM

This description has an Appendix A.

The present invention is directed on a tool with a tool body and a protective layer system, wherein the layer system comprises at least one layer of MeX, wherein

- Me comprises titanium and aluminum,

- X is at least one of nitrogen and of carbon.

Definition:

- The term Q_I is defined as the ratio of the diffraction intensities I(200) to I(111), assigned respectively to the (200) and (111) plains in the X ray diffraction of a material using the θ-2θ method. Thus, there is valid $Q_I = I(200)/I(111)$. The intensity values were measured with the following equipment and with the following settings:

Siemens Diffractometer D500

Power:	Operating voltage:	30 kV
	Operating current:	25 mA
Aperture Diaphragms:	Diaphragm position I:	1°
	Diaphragm position II:	0.1°
Detector Diaphragm:	Soller slit	
Time constant:	4 s	
2ϑ angular speed:	0.05°/min	
Radiation:	Cu-Kα(0.15406 nm)	

When we refer to "measured according to MS" we refer to this equipment and to these settings. Thereby, all quantitative re-

sults for Q_I and I throughout this application have been measured by MS.

- We understand by "tool body" the uncoated tool.

- We understand under "hard material" a material with which tools which are mechanically and thermally highly loaded in operation are coated for wear resistance. Preferred examples of such materials are referred to below as MeX materials.

It is well-known in the tool-protecting art to provide wear resistant layer systems which comprise at least one layer of a hard material, as defined by MeX.

The present invention has the object of significantly improving the lifetime of such tools. This is resolved by selecting for said at least one layer a Q_I value, for which there is valid

$$Q_I \leq 2$$

and wherein the tool is a solid carbide end mill or a solid carbide ball nose mill or a cemented carbide gear cutting tool. Further, the value of I(111) is higher by a factor of at least 20 than the intensity noise average level as measured according to MS.

According to the present invention it has been recognised that the Q_I values as specified lead to an astonishingly high improvement of wear resistance, and thus of lifetime of a tool, if such a tool is of the kind as specified.

Up to now, application of a wear resistant layer system of MeX hard material was done irrespective of interaction between tool body material and the mechanical and thermal load the tool is

subjected to in operation. The present invention thus resides on the fact that it has been recognised that an astonishing improvement of wear resistance is realised when selectively combining the specified Q_I value with the specified kind of tools, thereby realising a value of I(111) higher by a factor of at least 20 than the average noise intensity level, both measured with MS.

The inventively reached improvement is even increased if Q_I is selected to be at most 1, and an even further improvement is realised by selecting Q_I to be at most 0.5 or even to be at most 0.2. The largest improvements are reached if Q_I is at most 0.1. It must be stated that Q_I may drop towards zero, if the layer material is realised with a unique crystal orientation according to a vanishing diffraction intensity I(200). Therefore, there is not set any lower limit for Q_I which is only set by practicability.

As is known to the skilled artisan there exists a correlation between hardness of a layer and stress therein. The higher the stress, the higher the hardness. Nevertheless, with rising stress, the adhesion to the tool body tends to diminish.

For the tool according to the present invention a high hardness is rather more important than the best possible adhesion. Therefore, the stress in the MeX layer is advantageously selected rather at the upper end of the stress range given below. These considerations limit in practice the Q_I value exploitable.

In a preferred embodiment of the inventive tool, the MeX material of the tool is titanium aluminum nitride, titanium aluminum carbonitride or titanium aluminum boron nitride, whereby

the two materials first mentioned are today preferred over titanium aluminum boron nitride.

In a further form of realisation of the inventive tool, Me of the layer material MeX may additionally comprise at least one of the elements boron, zirconium, hafnium, yttrium, silicon, tungsten, chromium, whereby, out of this group, it is preferred to use yttrium and/or silicon and/or boron. Such additional element to titanium and aluminum is introduced in the layer material, preferably with a content i, for which there is valid

$$0.05 \text{ at.\%} \leq i \leq 60 \text{ at.\%},$$

taken Me as 100 at%.

A still further improvement in all different embodiments of the at least one MeX layer is reached by introducing an additional layer of titanium nitride between the MeX layer and the tool body with a thickness d, for which there is valid

$$0.05 \text{ µm} \leq d \leq 5 \text{ µm}.$$

In view of the general object of the present invention, which is to propose the inventive tool to be manufacturable at lowest possible costs and thus most economically, there is further proposed that the tool has only one MeX material layer and the additional layer which is deposited between the MeX layer and the tool body.

Further, the stress σ in the MeX is preferably selected to be

$$2 \text{ GPa} \leq \sigma \leq 8 \text{ GPa},$$ thereby most preferably

within the range

$$4 \text{ GPa} \leq \sigma \leq 6 \text{ GPa} .$$

The content x of titanium in the Me component of the MeX layer is preferably selected to be

$70 \text{ at } \% \geq x \geq 40 \text{ at } \%$, thereby in a further preferred embodiment within the range

$$65 \text{ at } \% \geq x \geq 55 \text{ at } \%.$$

On the other hand, the content y of aluminum in the Me component of the MeX material is preferably selected to be

$30 \text{ at } \% \leq y \leq 60 \text{ at } \%$, in a further preferred embodiment even to be

$$35 \text{ at } \% \leq y \leq 45 \text{ at } \%.$$

In a still further preferred embodiment, both these ranges, i.e. with respect to titanium and with respect to aluminum are fulfilled.

The deposition, especially of the MeX layer, may be done by any known vacuum deposition technique, especially by a reactive PVD coating technique, as e.g. reactive cathodic arc evaporation or reactive sputtering. By appropriately controlling the process parameters, which influence the growth of the coating, the inventively exploited Q_I range is realised.

To achieve excellent and reproducible adhesion of the layers to the tool body a plasma etching technology was used, as a preparatory step, based on an Argon plasma as described in Appendix A, which document is integrated to this description by reference, with respect to such etching and subsequent coating.

This document accords with the US application No. 08/710 095 of the same inventor (two inventors!) and applicant as the present application.

Examples 1

An arc ion plating apparatus using magnetically controlled arc sources as described in Appendix A was used operated as shown in table 1 to deposit the MeX layer as also stated in table 1 on solid carbide end mills with a diameter of 10 mm, z = 6. The thickness of the MeX layer deposited was always 3 µm. Thereby, in the samples Nr. 1 to 5, the inventively stated Q_I values where realised, whereas, for comparison, in the samples number 6 to 10 this condition was not fulfilled. The I(111) value was always significantly larger than 20 times the noise average value, measured according to MS. The coated end mills were used for milling under the conditions stated below to find the milling distance attainable up to attaining an average width of flank wear of 0.20 mm. The resulting milling distance according to the lifetime of such tools is also shown in table 1.

Test cutting conditions:

- Tool:	Solid carbide end mill,
	dia. 10 mm, z = 6
- Material being cut:	AISI D2 (DIN 1.2379)
- Cutting parameters:	v_c = 20m/min
	f_t = 0.031 mm
	a_p = 15 mm

a_e = 1 mm

climb milling, dry

It is clearly recognisable from table 1 that the end mills, coated according to the present invention, are significantly more protected against delamination and wear than the end mills coated according to the comparison conditions.

(Table 1)

Sample No.		Coating Conditions		Film $x = 0.5$; $Y = 0.5$	Q_I = I(200)/I(111)	Cutting Distance (m)	Residual Compressive Stress (-GPa)
		Bias Voltage (-V)	N_2-pressure (mbar)				
present Invention	1	200	3.0×10^{-2}	$Ti_xAl_y)N$	0.1	28m (normal wear 0.2 mm)	5.7
	2	150	2.0×10^{-2}	$(Ti_xAl_y)N$	1.0	23m (normal wear 0.2 mm)	4.0
	3	100	1.0×10^{-2}	$(Ti_xAl_y)N$	0.9	22m (normal wear 0.2 mm)	4.5
	4	100	2.0×10^{-2}	$(Ti_xAl_y)N$	2.0	18m (normal wear 0.2 mm)	2.9
	5	100	0.5×10^{-2}	$(Ti_xAl_y)N$	1.0	25m (normal wear 0.2 mm)	4.8
Comparison	6	20	3.0×10^{-2}	$(Ti_xAl_y)N$	8.0	3m (chipping and delamination)	1.3
	7	40	3.0×10^{-2}	$(Ti_xAl_y)N$	8.1	2m (chipping and delamination)	1.6
	8	40	2.0×10^{-2}	$(Ti_xAl_y)N$	4.2	4m (chipping and delamination)	2.0
	9	70	3.0×10^{-2}	$(Ti_xAl_y)N$	5.2	8m (normal wear 0.2 mm)	1.8
	10	70	2.5×10^{-2}	$(Ti_xAl_y)N$	3.0	10m (normal wear 0.2 mm)	2.3

Examples 2:

The apparatus as used for coating according to Example 1 was also used for coating the samples Nr. 11 to 20 of table 2. The tools coated and the test conditions were identical to Example 1. The thickness of the layers is indicated in table 2.

It may be seen that in addition to the coating according to Example 1 there was applied an interlayer of titanium nitride between the MeX layer and the tool body and an outermost layer of the respective material as stated in table 2. The condition with respect to I(111) and average noise level, measured according to MS was largely fulfilled.

It may be noted that provision of the interlayer between the MeX layer and the tool body already resulted in a further improvement. An additional improvement was realised by providing an outermost layer of one of the materials titanium carbonitride, titanium aluminum oxinitride and especially with an outermost layer of aluminum oxide. Again, it may be seen that by realising the inventively stated Q_I values with respect to the comparison samples number 16 to 20, a significant improvement is realised.

The outermost layer of aluminum oxide of 0.3 μm thickness, was formed by plasma CVD.

As stated above, the coated end mills were tested under the same cutting conditions as those of Example 1, Q_I was measured according to MS.

(Table 2)

Sample No.		Interlayer (μm)	TiAl Layer $x = 0.5$; $y = 0.5$	Outermost Layer	I(200)/I(111)	Cutting Distance (m)
Present Invention	11	TiN (0.4 μm)	$(Ti_xAl_y)N$ (2.6 μm)	-	1.5	30m (normal wear 0.2 mm)
	12	TiN (3.0 μm)	$(Ti_xAl_y)N$ (2.3 μm)	TiCN (0.3 μm)	1.2	32m (normal wear 0.2 mm)
	13	TiN (1.5 μm)	$(Ti_xAl_y)N$ (2.55 μm)	TiCN (0.3 μm)	0.5	38m (normal wear 0.2 mm)
	14	TiN (0.8 μm)	$(Ti_xAl_y)N$ (2.4 μm)	(TiAl)NO (0.3 μm)	0.5	35 m (normal wear 0.2 mm)
	15	TiCN (0.3 μm)	$(Ti_xAl_y)N$ (2.4 μm)	Al_2O_3 (0.3 μm)	2.0	39 m (normal wear 0.2 mm)
Comparison	16	TiN (1.0 μm)	$(Ti_xAl_y)N$ (2.0 μm)	-	3.0	12m (normal wear 0.2 mm)
	17	TiN (1.5 μm)	$(Ti_xAl_y)N$ (2.3 μm)	TiCN (0.3 μm)	5.8	8m (chipping)
	18	TiN (0.4 μm)	$(Ti_xAl_y)N$ (2.3 μm)	TiCN (0.3 μm)	4.5	14m (normal wear 0.2 mm)
	19	TiN (0.8 μm)	$(Ti_xAl_y)N$ (2.4 μm)	(TiAl)NO (0.3 μm)	8.2	10m (normal wear 0.2 mm)
	20	TiN (0.3 μm)	$(Ti_xAl_y)N$ (2.4 μm)	Al_2O_3 (0.3 μm)	12.3	10m (normal wear 0.2 mm)

Example 3:

Again, solid carbide end mills were coated with the apparatus of Example 1 with the MeX layer as stated in table 3, still fulfilling the Q_I conditions as inventively stated and, by far, the condition of I(111) with respect to average noise level, measured according to MS. Thereby, there was introduced one of zirconium, hafnium, yttrium, silicon and chromium, with the amount as stated above, into Me.

The coated end mills were kept in an air oven at 750°C for 30 min. for oxidation. Thereafter, the resulting thickness of the oxide layer was measured. These results are also shown in table 3. For comparison, inserts coated inventively with different Me compounds of the MeX material were equally tested. It becomes evident that by adding any of the elements according to samples 23 to 32 to Me, the thickness of the resulting oxide film is significantly reduced. The best results with respect to oxidation were realised by adding silicon or yttrium.

It must be pointed out, that it is known to the skilled artisan, that for the MeX material wear resistant layers there is valid: The better the oxidation resistance and thus the thinner the resulting oxide film, the better the cutting performance.

(Table 3)

	Sample No.	Layer Composition	w	x	y	z	Thickness of Oxide Film (μm)
Present Invention	21	$(Ti_xAl_yY_z)N$		0.48	0.5	0.02	0.7
	22	$(Ti_xAl_yCr_z)N$		0.48	0.5	0.02	0.9
	23	$(Ti_xAl_yZr_z)N$		0.48	0.5	0.02	0.7
	24	$(Ti_xAl_yY_z)N$		0.25	0.5	0.25	0.1
	25	$(Ti_xAl_yZr_z)N$		0.25	0.5	0.25	0.5
	26	$(Ti_xAl_yW_z)N$		0.4	0.5	0.1	0.8
	27	$(Ti_xAl_ySi_z)N$		0.4	0.5	0.1	0.1
	28	$(Ti_xAl_ySi_z)N$		0.48	0.5	0.02	0.2
	29	$(Ti_xAl_yHf_z)N$		0.4	0.5	0.1	0.9
	30	$(Ti_xAl_yY_zSi_w)N$	0.1	0.3	0.5	0.1	0.05
Comparison	31	$(Ti_xAl_y)N$		0.4	0.6		1.8
	32	$(Ti_xAl_yNb_z)N$		0.4	0.5	0.1	2.5
	33	$(Ti_xAl_yTa_z)N$		0.4	0.5	0.1	3.3

Example 4:

Again, the apparatus and coating method according to Example 1 was used. Solid carbide end mills with a diameter of 10 mm with 6 teeth were coated with a 3.0 µm MeX layer. There was provided a titanium nitride interlayer with a thickness of 0.08 µm between the MeX and the tool body. Test conditions for the end mills were:

Tool:	Solid carbide end mill, dia. 10 mm
	z = 6
Material:	AISI D2 (DIN 1.2379)
	60 HRC
Cutting parameters:	v_c = 20 m/min
	f_t = 0.031 mm
	a_p = 15 mm
	a_e = 1 mm
	Climb milling, dry

The solid carbide end mills were used until an average width of flank wear of 0.20 mm was obtained. The result is shown in table 4. Again, the I(111) to noise condition, measured with MS, was clearly fulfilled for sample No. 35, for sample No. 34 the I(200) to noise condition was fulfilled.

(Table 4)

		Bias Voltage (-V)	N_2- Pressure (mbar)	Arc current (A)	inter-layer	layer	x	y	Q_1	Residual Stress (GPa)	Cutting distance (m)
Comparison	34	40	3.0×10^{-2}	200	TiN 0.08 µm	$(Ti_xAl_y)N$	0.6	0.4	5.0	2.2	17m
Present Invention	35	150	1.0×10^{-2}	200	TiN 0.08 µm	$(Ti_xAl_y)N$	0.6	0.4	0.05	4.7	32m

Example 5:

The apparatus and coating method according to Example 1 was used.

Solid carbide ball nose mills were coated with 3.1 µm MeX and a TiN interlayer with the thickness 0.07 µm. The coated tools were tested with milling a hardened mold steel.

Test conditions:

Tool:	Solid carbide ball nose mill J97 (Jabro), R4 (∅ 8 x 65 mm)
Material:	Mold steel H 11 (DIN 1.2343), HRC 49.5
Cutting parameters:	v_c = 220 m/min
	a_p = 0.5 mm
	no coolant

The tool life was evaluated in minutes.

(Table 5)

		Bias Voltage (-V)	N_2 Pressure (mbar)	Arc current (A)	Inter-layer	layer	x	Y	Q_I	Residual Stress (GPa)	Tool life (m)
Present Invention	1	150	1.0×10^{-2}	200	TiN 0.07 µm	$(Ti_xAl_y)N$	0.6	0.4	0.04	5.1	198 m
Comparison	2	40	3.0×10^{-2}	200	TiN 0.07 µm	$(Ti_xAl_y)N$	0.6	0.4	5.7	1.8	127 min

In Fig. 1 there is shown, with linear scaling a diagram of nitrogen partial pressure versus bias voltage of the tool body as applied for reactive cathodic arc evaporation as the reactive PVD deposition method used to realise the Examples which were discussed above.

All the process parameters of the cathodic arc evaporation process, namely

- arc current;
- process temperature
- deposition rate
- evaporated material
- strength and configuration of magnetic field adjacent the arc source
- geometry and dimensions of the process chamber and of the workpiece tool to be treated

were kept constant. The remaining process parameters, namely partial pressure of the reactive gas - or total pressure - and bias voltage of the tool body to be coated as a workpiece and with respect to a predetermined electrical reference potential, as to the ground potential of the chamber wall, were varied.

Thereby, titanium aluminum nitride was deposited. With respect to reactive gas partial pressure and bias voltage of the tool body, different working points were established and the resulting Q_I values at the deposited hard material layers were measured according to MS.

It turned out that there exists in the diagram according to fig. 1 an area P, which extends in a first approximation linearly from at least adjacent the origin of the diagram coordinates, wherein the resulting layer leads to very low XRD

intensity values of I(200) and I(111). It is clear that for exactly determining the limits of P, a high number of measurements will have to be done. Therein, none of the I(200) and I(111) intensity values is as large as 20 times the average noise level, measured according to MS.

On one side of this area P and as shown in fig. 1 Q_I is larger than 1, in the other area with respect to P, Q_I is lower than 1. In both these areas at least one of the values I(200), I(111) is larger than 20 times the average noise level, measured according to MS.

As shown with the arrows in fig. 1, diminishing of the partial pressure of the reactive gas - or of the total pressure if it is practically equal to the said partial pressure - and/or increasing of the bias voltage of the tool body being coated, leads to reduction of Q_I. Thus, the inventive method for producing a tool which comprises a tool body and a wear resistant layer system, which latter comprises at least one hard material layer, comprises the steps of reactive PVD depositing the at least one hard material layer in a vacuum chamber, thereby preselecting process parameter values for the PVD deposition process step beside of either or both of the two process parameters, namely of partial pressure of the reactive gas and of bias voltage of the tool body. It is one of these two parameters or both which are then adjusted for realising the desired Q_I values, thus, and according to the present invention, bias voltage is increased and/or partial reactive gas pressure is reduced to get Q_I values, which are, as explained above, at most 2, preferably at most 1 or even at most 0.5 or even at most 0.2. Most preferred is $Q_I \leq 0.1$. Beside the inventively exploited Q_I value, in this "right hand" area, with respect to

P, I(111) is larger, mostly much larger than 20 times the average noise level of intensity, measured according to MS.

In fig. 2 a typical intensity versus angle 2θ diagram is shown for the titanium aluminum nitride hard material layer deposited in the $Q_I \geq 1$ region of fig. 1, resulting in a Q_I value of 5.4. The average noise level N* is much less than I(200)/20. Measurement is done according to MS.

In Fig. 3 a diagram in analogy of that in fig. 2 is shown, but the titanium aluminum nitride deposition being controlled by bias voltage and nitrogen partial pressure to inventively result in a $Q_I \leq 1$. The resulting Q_I value is 0.03. Here again the I(111) value is larger than 20 times the average noise level of intensity, both measured according to MS.

Please note that in fig. 1 the respective Q_I values in the respective regions are noted at each working point measured (according to MS).

In fig. 4 a diagram in analogy to that of the figs. 2 and 3 is shown for working point P_1 of fig. 1. It may be seen that the intensities I(200) and I(111) are significantly reduced compared with those in the area outside P. None of the values I(200) and I(111) reaches the value of 20 times the noise average level N*.

Thus, by simply adjusting at least one of the two Q_I-controlling reactive PVD process parameters, namely of reactive gas partial pressure and of workpiece bias voltage, the inventively exploited Q_I value is controlled.

In fig. 1 there is generically shown with $\partial Q_I < 0$ the adjusting direction for lowering Q_I, and it is obvious that in opposite direction of adjusting the two controlling process parameters, and increase of Q_I is reached.

Process and apparatus for workpiece coating

The present invention relates to a coating arrangement according to the generic specification of claim 1 as well as a process for coating workpieces according to the generic specification of claim 14.

In many known vacuum treatment processes, cleaning of the workpiece surface is performed prior to vacuum coating. In addition, the workpieces may be heated to the desired temperature before or after the cleaning step. Such steps are principally needed to ensure adequate bonding strength of the coating to be deposited. This is especially important in applications where workpieces, and tools in particular, are to be coated with a wear protection coating. On tools such as drills, milling cutters, broaches and shaping dies such coatings are subjected to very high mechanical and abrasive stress. An extremely good bond with the substrate is, therefore, essential for useful and economical use. A proven method for pre-treating such tools is heating with electron bombardment, and etching by means of ion etching, for example, sputter etching. Heating by means of electron bombardment from a plasma discharge is known, for example, from DE 33 30 144.

A plasma discharge path can also be used for creating heavy noble gas ions, for example, argon ions, which are accelerated from this plasma toward the workpiece or the substrate on which they cause sputter etching as described in DE 28 23 876.

In addition to sputter etching another known technique is to operate plasma discharges with additional reactive gases and to etch the workpieces chemically, however, also process techniques combining reactive etching and sputter etching are feasible. The objective of all these pre-treatment processes is to prepare the workpiece surface in such a way that the subsequently deposited coating adheres well to the substrate.

For plasma generation the aforementioned arrangements use a low-voltage arc discharge that is arranged in the central axis of the apparatus whereas the workpieces are arranged at a certain distance around this arc along a cylindrical surface. The coating is subsequently deposited by means of thermal evaporation or sputtering. Depending on the process management, an additional ion bombardment is generated during the coating through a corresponding substrate bias, a technique which is known as ion plating. The advantage of this arrangement is that large ion currents with small particle energy can be drawn from the low-voltage arc which affords gentle treatment of the workpiece. The disadvantage is, however, that the workpieces must be arranged in a zone defined radially to the discharge and that as a rule they must be rotated round the central axis as well as their own axis in order to achieve uniform and reproducible results.

Another disadvantage is that due to the relatively narrow admissible cylindrical processing band width either the processable workpiece size is limited or the batch size is limited for a large number of small workpieces which severely limits the cost-effectiveness of the known arrangements. This limitation is due to the fact that the low-voltage arc discharge which centrally penetrates the process chamber requires a certain dimension for itself. In order to produce good and reproducible results, the workpieces must have a suitable distance from the discharge which means that a large portion of the central process chamber space cannot be utilized.

Also known are sputtering arrangements with so-called diode discharges. Such diode discharges are operated with high voltages of up to 1000 Volt and even higher. Diode etching devices have proven to be unsuited to applications with demanding requirements. On the one hand the achievable etching rates and consequently the efficiency is low, and on the other hand these high voltages can produce defects on sensitive substrates. In particular workpieces that require three-dimensional processing such as tools cannot be readily processed by such an arrangement. Tools, for example, are

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designed with a number of fine cutting edges on which such discharges tend to concentrate, with the result that uncontrolled effects such as overheating and even destruction of the functional edge can occur on such fine edges and points.

In the patent application DE 41 25 365 an approach for solving the aforementioned problem is described. It assumes that the coating is deposited by means of a so-called arc evaporation process. In order to produce well-bonding coatings with such evaporators, the arc of the evaporator itself was used prior to the actual coating in such a way that the ions produced in the arc, particularly the metal ions, are accelerated out of the evaporation target toward the workpieces by means of a negative acceleration voltage of typically > 500 Volt, but often also in the range of 800 to 1000 volt so that more material is sputtered off the workpiece than deposited. After this etching process the evaporator is operated as a coating source. The description mentions that in the usual processes based on the arc coating technology such high voltages are essential for producing well-adhering coatings through the arc evaporation process.

To prevent the problem of overheating or etching on uneven mass distribution or on fine workpiece geometries, the description proposes to operate, in addition to the arc plasma, an auxiliary discharge path with high voltage that causes supplementary ionization which is coupled to the evaporation arc. An additional DC source causes ions to be extracted from the plasma and accelerated to the workpiece and thereby produce the desired etching effect. An additional anode with another discharge path operated from a separate power supply is envisioned for increasing the effect. During the etching process the arc evaporator is operated with a closed shutter so that the substrate is shielded from the direct effect of the evaporator, thereby preventing so-called droplets on the substrate.

The disadvantage of the above arrangement is that it also requires a high voltage, that only limited processing homo-

geneities are achievable, and that through the coupling of the different plasma paths also the adjustment capabilities in the operating environments are limited. In addition this arrangement is very complicated and consequently costly to build and operate which seriously impairs the economy of a production system. The utilization of voltages in excess of 1000 Volt requires additional safety precautions.

Systems that are based on the current technology are not well suited to high throughputs if also high processing quality is required. Systems that accommodate coating widths of up to 1000 mm and more can be built only with great difficulty, if at all.

The purpose of the present invention is to eliminate the aforementioned disadvantages of the current technology, in particular by creating a coating arrangement and by proposing a process that is suitable for depositing well-adhering coatings on a large number of workpieces, or on individual large workpieces with uneven mass distribution, without damaging the fine structures but with the desired homogeneity and the required highly economical processing rate.

This is achieved by designing the process arrangement mentioned at the beginning in accordance with the characterizing portion of claim 1, and by the coating process designed according to the characterizing portion of claim 14.

Accordingly the workpiece surface to be coated is exposed to a plasma source designed as a hot cathode low-voltage arc discharge arrangement by transporting it transversely to the linear extent of the latter's discharge path. The workpiece is connected to a negative voltage so that ions are extracted from the arc discharge and accelerated to the workpiece, causing the latter to be sputter etched. Subsequently the workpiece is coated from the same side from which the low-voltage arc discharge was effective.

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The preferred design variants of the coating arrangement conforming to the invention are described in the subsidiary claims 2 to 13, and the preferred design variants of the process in claims 14 to 17.

Etching with a hot cathode low-voltage arc discharge arrangement as the ion source is particularly advantageous because such arc discharges can be operated with discharge voltages of < 200 Volt which means that this process is not afflicted by the disadvantages of high-voltage etching. Etching with low-voltage arc discharges is also particularly harmless to the workpiece, that is, the fine structures on larger workpieces such as cutting edges are adversely affected neither by thermal overload nor edge rounding caused by high-energy ion bombardment.

Despite the relatively low discharge voltage in the working range of 30 to 200 Volt DC, but preferably within the range of 30 to 120 Volt, a very high discharge current of a few 10 to a few 100 ampere, preferably from 100 to 300 ampere, is feasible. This means that this type of discharge is able to produce a very high ion current at low energy. Due to the high ion current available, a high etching rate can be achieved on the substrate at a relatively low acceleration voltage, and as has been mentioned before, with gentle treatment of the workpiece. The extraction voltage or the acceleration voltage on the substrate is within the range of -50 Volt to -300 Volt, preferably within the range of -100 Volt to -200 Volt. The ion current drawn to the workpieces achieves values of 5 to 20 ampere, with a preferred working range from 8 to 16 ampere. The processing width for the workpiece or workpieces can be up to 1000 mm. With a somewhat more elaborate equipment design also larger processing widths are feasible. The achievable values depend not only on the operating values for the arc discharge but also on their geometric arrangement relative to the workpiece, as well as on the selected working pressure. Typical working pressures are of the order of 10^{-3} mbar. For operating the

arc discharge a noble gas is used as the working gas, preferably a heavy noble gas such as argon.

In the past, low-voltage arc discharge arrangements were rotation symmetrical which means that the arc discharge was arranged in the center and the workpieces were rotated around this arc discharge located in the central axis. The assumption was that the rotation symmetrical arrangement with the centrally arranged arc discharge would offer the best possible result with respect to uniformity and speed of the etching operation. Surprisingly it has been shown, however, that the asymmetrical arrangement proposed by the invention is overall much more advantageous than the aforementioned rotation symmetrical arrangement. With a rotation symmetrical arrangement with the arc discharge in the central axis the placement of large volume workpieces is restricted toward the center by the arc discharge itself. In addition such workpieces have to be rotated not only around the central axis but also around their own axis so that after the etching process the etched workpiece surfaces can be coated immediately with the coating sources arranged on the chamber wall. Only in this way is adequate distribution of the etching process and the coating thickness ensured.

It has also been shown that the distance of the workpiece from the arc discharge is more critical in a rotation symmetrical arrangement than in an asymmetrical arrangement in which the workpiece is exposed only from one side toward the arc discharge.

In the apparatus according to the invention it is possible to pass large-volume workpieces in front of the arc discharge without additional rotation, with the result that the size of the process chamber can be kept within reasonable limits and the handling of heavy workpieces is greatly simplified. This has a significant influence on the economy of production systems. The arrangement according to the invention is advantageous not only for large-volume workpieces but it is also possible to accommodate and simultaneously

process a correspondingly large number of smaller work-pieces.

Another advantage of the arrangement according to the invention is that the etching apparatus no longer has to be constructed as an integral part of the process chamber because it needs to be arranged only in the area of the process chamber wall which means that it can be arranged as an elongated, smaller discharge chamber on the latter's outer wall so that far greater freedom is achieved in the design of the process chamber. It has even been found that this arrangement is far less critical with respect to the distance between the arc discharge and the workpiece surface, which means that higher reproducibility of the results is achieved with larger spacing variations that typically occur with larger workpieces. The total ion current that can be extracted from the arc discharge still reaches advantageously high values and can be concentrated fully on the workpieces, thereby producing the desired high etching rates. The actual separation of the low-voltage arc discharge or the plasma source from the process chamber or from the treatment zone also affords a higher degree of freedom in the design of this source and consequently a much more flexible adaptation of the source design to the process requirements than is the case with the integral rotation-symmetrical arrangement with discharge in the central axis of the equipment.

For depositing a well-bonding coating after the etching process, one or more additional evaporation sources acting from the same side are arranged on the process chamber wall. Particularly suited are sources that can be arranged in such a way that, like the elongated low-voltage discharge, they coat the workpieces transported in front of them across a correspondingly elongated area. Suited are sources such as sputtering sources or arc evaporation sources. Practice has shown that so-called cathodic spark evaporators or arc evaporators are particularly suited because well-bonding coatings can be economically produced by these and the preceding etching process. Test tools processed through this

arrangement achieved a useful life that was significantly and reproducibly longer than achieved by known arc evaporated coatings with preceding high-voltage etching. For example, the useful life of cutting tools such as milling cutters was improved by a factor of at least 1.5; in particularly favorable cases even by a multiple over conventional techniques. In addition a very homogenous etch distribution was achieved which is far less dependent on the workpiece geometry and also allows mixing of different substrates in a batch.

With the proposed arrangement it is also easily possible to implement processes not only with noble gases but also with chemically active gases because the low-voltage arc discharge activates gases such as N_2, H_2 very well. Unwanted parasitic discharges produced by insulating surfaces can be easily controlled with the low-voltage discharge. The low-voltage arc discharge is preferably operated with a separate cathode chamber or ionization chamber that accommodates a hot cathode and communicates with the discharge chamber or the process chamber only through a small opening. The gases are preferably admitted via this cathode chamber. This results in a certain gas separation between the process chamber and the coating sources which reduces or eliminates the problem of target contamination. With this arrangement it is also possible to perform activation on the workpiece with different process gases during the actual coating phase. The desired working conditions can be established by choosing a corresponding negative or even positive voltage on the workpiece.

As the workpieces generally have to be passed in front of the sources several times during a process step in order to achieve the necessary etching depth or coating thickness as well as uniform and reproducible treatment, it is advantageous to design the apparatus in such a way that the workpieces can be rotated around a central axis and to arrange the sources on the chamber wall in such a way that they all work from the outside toward the inside. In this case a very

large workpiece can be arranged for processing in such a way that it rotates on its central axis. In the same space, however, also a large number of small workpieces, even of different size, can be arranged on a holder and passed across the sources while rotating around this central axis in order to achieve homogenous results. Such an arrangement is particularly compact and easy to build which is essential for an economical process.

The plasma source or the low-voltage arc discharge is preferably arranged on the process chamber wall, transversely to the transport direction. The low-voltage arc discharge device can, for example, and preferably be arranged in a box-like attachment, here in the form of a discharge chamber, which is connected to the process chamber by a long narrow opening in such a way that the low-voltage arc is arranged directly opposite the workpiece(s) or the zone to be processed. The low-voltage arc discharge is generated by an electrically heated or thermionic emission cathode and an anode arranged at a certain distance. A corresponding discharge voltage is applied to this anode, causing an arc current to be drawn. This discharge features a gas inlet port through which the arc discharge is supplied with the working gas. This arrangement is preferably operated with a noble gas such as argon, but as has been mentioned above, also reactive gases can be added. The size of the discharge path should be at least 80% of the treatment zone width and be positioned relative to the treatment zone in such a way that the desired treatment distribution or homogeneity can be attained. To achieve the corresponding sputter etching on the workpiece, the latter or the workpiece holder is operated with a negative voltage relative to the arc discharge arrangement. Depending on the process, such as in reactive processes during the coating, the arrangement can also be operated without such a voltage or even with a positive voltage, that is, with electron bombardment. Aside from a DC voltage also a medium or high-frequency AC voltage can be used, and also superposition of DC on AC is feasible. The DC voltage can also be pulsating, and it is possible to super-

pose only part thereof on the AC supply. With such a supply it is possible to control certain reactive processes. It also can in particular avoid or prevent parasitic arcs if dielectric zones exist or are formed on the equipment and the workpiece surfaces.

The desired distribution with respect to the processing zone can be set via the length of the discharge and its location. Another parameter for controlling the distribution is the plasma density distribution along the arc discharge. This distribution can e.g. be influenced with the aid of additional magnetic fields which are arranged in the area of the discharge chamber. For the setting and correction of the process parameters, permanent magnets are positioned along the discharge chamber. Better results are achieved, however, if the discharge path is operated with additional, separately powered anodes which are arranged along the discharge path in accordance with the distribution requirements. With such an arrangement even the distribution curve can be influenced to a certain degree. Preferred is, therefore, the arrangement without correction magnets and with more than one anode along the discharge path. However, it is also possible to combine this preferred arrangement with additional correction magnets. Additional anodes can be readily operated in combination with a single cathode. It is advantageous, however, to have an emission cathode opposite each anode in order to achieve optimum decoupling of these circuits which in turn improves the controllability.

The thermionic emission cathode is preferably arranged in a separate, small cathode chamber which communicates with the discharge chamber through a small opening. This cathode chamber is preferably equipped with an inlet port for noble gas. If desired also reactive gases can be admitted via this gas inlet. Preferably, reactive gases are not admitted into the cathode chamber but, for example, into the discharge chamber. Through the opening in the cathode chamber the electrons are drawn to the anode or anodes so that the gas which is a least partially ionized also emerges from this

opening. The process chamber is preferably designed in such a way that the central axis around which the workpieces are rotating, is arranged vertically. The cathode or the cathode chamber is preferably arranged above the anode. In the cathode chamber the exit opening is preferably arranged downward. These arrangements simplify the entire handling of the system and help to avoid problems that can be caused by particle formation.

In addition to the low-voltage arc discharge arrangement the process chamber is equipped with at least one additional source, preferably in the form of an arc evaporator. These sources act radially in the same direction from the outside toward the central axis or the processing zone. It is advantageous if the low-voltage arc discharge is arranged before the coating source with respect to the transport direction. An arc evaporator, like the arc discharge arrangement, usually has a linear extent that is transverse to the transport direction so that the entire processing zone can be coated with the desired homogeneity. In the proposed coating arrangement several round arc evaporators are preferably used which are distributed along the chamber wall in such a way that the desired homogeneity is achieved. The advantage is that the high power consumption of the evaporator can be split up and that coating thickness distribution can be better controlled or to a certain degree be even adjusted by means of the power supply. In this way exceptionally high coating rates can be achieved which results in high economy. For example, a process for tools, particularly shaping dies, would be configured as follows:

Process example

The system configuration corresponds to illustrations 2 and 3. The tools are not rotated around their own axis but only passed in front of the sources by rotating the workpiece holder around its central axis. A coating zone with a width b of 1000 mm and a diameter d of 700 mm is formed, within which the workpieces are arranged. The process chamber has a diameter of 1200 mm and a height of 1300 mm.

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Etching parameters:

Low-voltage arc current	I_{LVA} = 200 A
Arc discharge voltage	U_{LVA} = 50 V
Argon pressure	$P_{Ar} = 2.0 \times 10^{-3}$ mbar
Etching current	I_{sub} = 12A
Etching time	t = 30 min
Etching depth	200 nm

Coating:

Current for each arc evaporator	I_{ARC} = 200 A
(8 evaporators with 150 mm diam. titanium targets)	
Arc discharge voltage	U_{ARC} = 20 V
Nitrogen pressure	$P_{N2} = 1.0 \times 10^{-2}$ mbar
Bias pressure	U_{Bias} = -100 V
Coating time	t = 45 min
Coating thickness TiN	6 µm

The process cycle time for one batch, including heating and cooling, is 150 min.

The voltage generation equipment for the negative acceleration voltage on the workpiece is usually operated with voltages of up to 300 Volt DC, but to protect the workpieces the voltage is preferably kept within the range of 100 to 220 Volt at which good etching rates are still feasible without defects. The low-voltage arc arrangement must be operated at least 10 cm away from the workpiece, but the distance should preferably be > 15 cm, or preferably within the range of 15 to 25 cm at which high rates with a good distribution are achieved.

The coating system according to this invention is particularly suitable for processing tools such as drills, milling cutters and shaping dies. The holders and the transport device are designed specifically for this type of tools. The present coating arrangement is generally able to achieve good results even if the workpieces to be coated are rotated only around the central axis of the equipment. In particu-

larly critical cases or if a very large number of small parts are to be loaded into the system, the rotation around the central axis can easily be supplemented in this design concept by adding additional rotating axes which in turn rotate around the central axis.

The invention is subsequently exemplified and schematically explained by means of the following illustrations:

Fig. 1 A coating arrangement with low-voltage discharge according to the conventional technology. (State of the art).

Fig. 2 Cross-section of a typical coating system according to the invention, with peripheral discharge chamber for low-voltage discharge

Fig. 3 Horizontal section of the system illustrated in Fig. 2.

Fig. 4a Cross-section of a part of the arrangement with discharge chamber for low-voltage arc discharge and multiple anodes arranged inside the chamber.

Fig. 4b Same as Fig. 4a but illustrated with separate cathode-anode discharge paths with the cathodes arranged in separate cathode chambers.

Fig. 4c Same as Figs. 4a and 4b, also with separate cathode-anode discharge paths, but with the cathodes arranged in a common cathode chamber.

Fig. 5 Service life comparison curves for tools coated with the conventional technology and the technology according to the present invention.

Fig. 1 illustrates a known workpiece coating arrangement. A vacuum chamber serves as process chamber 1 for accommodating a low-voltage arc discharge 18 which runs in the center of

vacuum chamber 1 along the latter's central axis 16 and to which magnetron sputtering sources 14 are flanged at the periphery from the outside to the chamber wall of process chamber 1. On the top of process chamber 1 there is a cathode chamber 2 that holds a thermionic hot cathode 3 which can be supplied via gas inlet 5 with the working gas, typically a noble gas like argon. For reactive processes also active gases can be added. Cathode chamber 2 communicates with process chamber 1 via a small hole in shutter 4. The cathode chamber is usually insulated from processing chamber by means of insulators 6. Shutter 4 is additionally insulated from the cathode chamber via insulator 6 so that shutter 4 can be operated on floating potential or auxiliary potential, as required. Anode 7 is arranged in the direction of the central axis 16 on the opposite side of cathode chamber 2. Anode 7 can have the form of a crucible and holds the material to be evaporated by the low-voltage arc discharge. During the etching process this evaporation option is not used; only ions are extracted from the low-voltage arc discharge and accelerated toward the workpieces in such a way that that the latter are sputter etched. For operating the low-voltage arc discharge 18 cathode 3 is heated with a heater supply unit so that cathode 3 emits electrons. Between cathode 3 and anode 7 there is an additional power supply 8 for operating the arc discharge. It usually produces a positive DC voltage on anode 7 in order to sustain the low-voltage arc 18. Between arc discharge 18 and the chamber wall of processing chamber 1, workpiece holders are arranged that hold the workpieces 11 which can be rotated around their vertical central axis 17 in order to achieve adequate process uniformity. The workpiece holders 10 are supported on an additional workpiece holder arrangement 12 which is equipped with a rotary drive by which these workpiece holders 10 are rotated around the central axis 16. In this type of equipment it is additionally necessary to focus the low-voltage arc discharge 18 via additional coils 13, for example in the form of Helmholz coils. It is evident that the workpieces 11 can be processed with the low-voltage arc discharge 18, that ion bombardment occurs when a nega-

tive voltage is applied to the substrate, and that electron bombardment is possible by applying a positive substrate voltage. In this way the workpieces can be pre-treated with the aid of a low-voltage arc discharge either by means of electron bombardment induced by heating, or through ion bombardment with sputter etching. Subsequently the workpiece 11 can be coated, either through evaporation of material from crucible 7 by means of the low-voltage arc, or through sputtering with magnetron sputter source 14 which is supplied by the power supply 15.

It is readily apparent that the mechanical assembly for substrate movement and the arrangement of the low-voltage arc discharge are rather complex in this layout. On the other hand the degree of freedom is severely restricted because the workpieces can only be arranged between the low-voltage arc discharge located in the center and the outer chamber wall. A system of this type is uneconomical to operate for large workpieces or large batch quantities.

An example of a preferred coating arrangement according to the invention is illustrated as a cross-section in Fig. 2. Process chamber 1 contains a workpiece holder 11 which is arranged in such a way that the workpieces can be rotated around the central axis 16 of the process chamber. The chamber is usually pumped down by the vacuum pumps 19 that maintain the working pressures required for the process steps. In the proposed arrangement a large workpiece 11 which extends beyond the central axis 16 can, for example, be arranged in process chamber 1 in such a way that this large workpiece 11 can be processed by the sources arranged on the process chamber wall. The zone available for loading the workpieces essentially fills process chamber 1 completely. In such an arrangement it is possible to position either a single large workpiece 11 or a large number of smaller workpieces which essentially fill the chamber volume.

The workpiece holder that rotates the workpieces 11 around the central axis 16, spans coating width b transversely to

the rotation direction. In the system according to the invention it is particularly advantageous that uniform and reproducible coating results can be achieved either across large coating widths b or across a large depth range that extends from the central axis 16 to the periphery of the coating width, that is, within the entire diameter D. Based on the known concentric arrangement according to the conventional technology in which these conditions were critical, it was not to be expected that an eccentric arrangement according to the present invention would produce better results. A large variety of workpiece geometries with fine edges and cutting edges can be handled in this large area without problems related to thermal stress or unwanted occurrence of arcs.

On the outer wall of the process chamber the etching and coating sources are positioned in such a way that they all act from the outside toward the workpieces. For the important preparatory sputter etching process the chamber wall features a slot shaped opening, the length of which corresponds at least to processing width b. Behind his opening 26 there is a box shaped discharge chamber 21 in which the low-voltage arc discharge 18 is generated. This low-voltage arc discharge 18 runs essentially parallel to processing width b and has an effective length which shall be at least 80% of processing width b. Preferably the discharge length should be equal to the processing width b or extend even beyond it.

The axis of arc discharge 18 has a distance d from the nearest processing zone, that is, the next workpiece section. This distance d shall be at least 10 cm, preferably 15 to 25 cm. This results in good process uniformity and a high sputtering rate can be maintained. In the lower part of discharge chamber 21, cathode chamber 2 is flanged on which communicates with discharge chamber 21 via orifice 4. Cathode chamber 2 contains a hot cathode 3 which is supplied via the heating power supply unit 9. This supply can be operated with AC or DC. Cathode chamber 2 features a gas inlet port 5 for supplying the working gas, normally a noble gas like

argon, or a noble gas - active gas mixture for certain reactive processes. It is also possible to admit working gases via process chamber 1 by means of auxiliary gas inlet 22. Active gases are preferably admitted directly into process chamber 1 via gas inlet 22.

In the upper part of discharge chamber 21 there is an electrode 7 which is designed as a anode. DC supply 8 is connected between cathode 3 and anode 7 in such a way that the positive pole is on anode 7 and a low-voltage arc discharge can be drawn. By applying a negative voltage to the workpiece holder or to the workpieces 11 with the aid of voltage generator 20 between the low-voltage arc discharge arrangement and the workpiece 11, argon ions are accelerated toward the workpieces so that the surface is sputter etched. This can be achieved with acceleration voltages of up to 300 Volt DC, but preferably with a voltage in the range of 100 Volt to 200 Volt to ensure gentle processing of the workpieces 11. The process uniformity can be set through appropriate positioning of cathode chamber 2, and by arranging anode 7 relative to processing width b of the workpieces to be processed in accordance with the process specifications. Another factor is the shape of anode 7. The latter can, for example, have either a flat, dished, or rectangular shape, or be designed as a tubular, cooled anode.

Fig. 3 shows a horizontal cross-section of the system based on Fig. 2. Shown is again the box-like discharge chamber 21 on the outer wall of process chamber 1 which communicates with the treatment zone through slot opening 26. Of course, several such discharge chambers can be arranged on a system as required, for example to further boost the processing effect. Also illustrated are the evaporation sources 23 which are flanged to the chamber wall. For example, magnetron sputter sources can be used as evaporation sources 23 but for achieving high processing speeds at low costs, so-called arc evaporation sources are preferably used. The advantage of this arrangement is that the arc evaporation sources 23 can be freely arranged from the outside in such a

way that through the distributed arrangement of multiple sources the desired coating homogeneity can be set and a high coating rate can be maintained. It has been shown that it is more advantageous not to use single, rectangular evaporation sources but several smaller, round sources that are arranged on the periphery of the system in accordance with the process requirements.

Fig. 4a illustrates another advantageous variant of the arrangement according to the invention in which cathode chamber 2 is located on the top of discharge chamber 21. The advantage is that the operating of the discharge path is least disturbed by particles which always occur in such a coating system. Also shown is a the possibility of subdividing the discharge path by using several anode-cathode circuits and making the intensity along discharge 1 adjustable. The main discharge is generated with power supply 8 between main anode 7 and cathode chamber 2. Additional ancillary discharges can be generated with auxiliary anodes 24 and auxiliary power supplies 25. In this way it is possible to adjust the power density of the discharge along the entire discharge path between anode 7 and cathode 2 locally and with respect to the intensity to the homogeneity requirements of the workpiece.

Fig. 4b shows an alternative arrangement. The anode-cathode paths can be kept completely apart, or even decoupled by using separate anodes 7, 24, separate cathodes 3, 3', and separate cathode chambers 2, 2'. Another version is illustrated in Fig. 4c in which two separates anodes 7, 24 are used, but a common cathode chamber 2 with two hot cathodes 3 and 3'.

Fig. 5 illustrates the test results of HSS finish milling cutters that were processed according to the invention (curve b) and the conventional technology (curve a). In both cases the milling cutters were given a 3.5 µm TiN coating. For the milling cutter according to the conventional technology (curve a) high-voltage etching was first performed in

the conventional manner whereas for the milling cutter represented by curve b the process according to the invention was used. The test conditions were as follows:

HSS finish milling cutter:	Diam. 16 mm
Number of teeth:	4
Test material:	42 CrMo4 (DIN 1.7225)
Hardness:	HRC 38.5
Infeed:	15 mm x 2.5 mm
Cutting speed	40 m/min
Feed per tooth	0.088 mm
Feed	280 mm/min

End of life: Spindle torque 80 (arbitrary unit)

The result shows clear improvements in the life of the tool treated according to the invention. An improvement by a factor of 1.5 or more is easily reached. Important is not only the extension of the tool life but also the flatter progressing of the torque curve which is indicative of the deterioration in tool quality toward the end of the tool life. In the example according to Fig. 5 this is clearly recognizable at a total milling depth of 15 m. Curve a which represents the conventional technology shows a sharp degradation in tool quality at a total milling depth of 15 m. This shows that the cutting quality achievable with the conventional technology has a greater variance across the entire tool life which means that it is not very consistent.

Systems built in accordance with the invention as illustrated in Figures 2 to 4 achieve far greater throughputs with the aforementioned high quality than system 1 which conforms to the conventional technology. Throughputs can easily be doubled or even increased by a factor of 3 to 5 which dramatically increases the economy.

"Appendix A"

Summary

For depositing hard coatings on high-performance tools that must be sputter etched before coating, the invention proposes to sputter etch the tools with a low-voltage arc discharge and to subsequently coat them from the direction they have been etched.

Patent claims

1. Coating arrangement for treating workpieces (11) with a vacuum process chamber (1) and a plasma source (18) arranged on the chamber, and with a coating source (23) arranged inside said chamber, and said chamber being equipped with a holding and/or transport device which defines a treatment zone (b) for positioning or passing the workpieces (11) in front of the sources, with said sources being arranged at a certain distance to the workpiece and acting from the same direction, characterized by a plasma source (18) designed as a hot cathode low-voltage discharge arrangement, the linear extent (l) of which in a direction transverse to the workpiece transport direction essentially corresponds to width (b) of the processing zone, and containing a device for generating an electrical field (20) between the arc discharge (18) and the workpiece (11).

2. Arrangement according to claim 1 in which the holding and transporting device for the workpieces (11) is arranged rotatable around central axis (16) of process chamber (1) and with sources (18, 23) arranged on the chamber wall in such a way that they all act radially from the outside in the direction of the central axis (16).

3. Arrangement according to claim 1 or 2 in which the plasma source of a discharge chamber (21) is arranged on the outer wall of chamber (1) where inside or on discharge chamber (21) a thermionic emission cathode (3), and at least 80% of the processing zone width away and along processing zone width (b), an anode (7) for gener-

ating a low-voltage arc discharge (18) is positioned and in which arrangement a noble gas port (5) in discharge chamber 21 with a voltage generator (20) is arranged between the anode-cathode circuit and workpiece (11) in such a way that the negative pole is on workpiece (11) so that the plasma source arrangement (2, 7, 18, 21) functions as a sputter etching device.

4. Arrangement according to one of the preceding claims in which at least one additional anode (24) extending along the plasma path at a certain distance from said plasma path is arranged between emission cathode (3) and anode (7) for adjusting the plasma density distribution along arc discharge (18).

5. Arrangement according to one of the preceding claims in which anode (7) and the additional anode (24) are connected to separate, adjustable power supplies (25), and featuring an opposite cathode (3) preferably for each anode (7, 25) which together with the corresponding anode (7,25) and the separate power supply (8, 25) forms it's own adjustable power circuit.

6. Arrangement according to one of the preceding claims in which the emission cathode (3) is arranged in a cathode chamber (2) separate from discharge chamber (21) and with said cathode chamber (2) communicating with the discharge chamber (21) via opening (4) through which the electrons can emerge, with the noble gas inlet port (5) preferably arranged on this cathode chamber (2).

7. Arrangement according to one of the preceding claims 2 to 6 in which process chamber (1) with its central axis (16) is arranged vertically, and cathode (3) or cathode chamber (2) is arranged above the anode (7, 24), and the opening (4) of cathode chamber (2) is preferably pointing downward.

8. Arrangement according to one of the preceding claims in which at least one coating source (23) which preferably consists of at least one arc evaporator (23) is arranged

on the process chamber wall next to the plasma source (18) which is located further ahead in the transport direction.

9. Arrangement according to one of the preceding claims in which the voltage generator (20) is designed for voltages of up to 300 V DC, preferably for 100 V to 200 V.

10. Arrangement according to one of the preceding claims in which the low-voltage arc discharge arrangement (18) is located at least 10 cm but preferably 15 to 25 cm away from workpiece (11).

11. Arrangement according to one of the preceding claims in which the holding and transport device is designed as a tool holder, particularly for drills, milling cutters and shaping dies.

12. Arrangement according to one of the preceding claims in which at least one magnetic field generator is arranged in or on discharge chamber (21) for adjusting the plasma density distribution.

13. Arrangement according to one of the preceding claims in which discharge chamber (21) has an opening along the full width (b) of the processing zone and with the opening facing the latter so that the processing zone is exposed to the arc discharge.

14. Process for at least partially coating workpieces (11) in a vacuum process chamber (1) with a plasma source (18) arranged on the process chamber and a coating source (23) and with a holding and/or transport device arranged in chamber (1) with said device determining a treatment zone (b) for positioning or passing the workpieces (11) in front of the sources (18, 23), where the sources act from the same side and are arranged at a certain distance from workpiece (11), and in which process the plasma source (18) generates a hot cathode low-voltage arc (18) in a direction transverse to the workpiece transport direction essentially at least across

80% of width (b) of the treatment zone and in which process a voltage is applied between the arc discharge and the workpiece for extracting charge carriers from the plasma so that they can be accelerated toward the substrate.

15. Process according to claim 14 in which the workpieces rotate preferably continuously around central axis (16) of a processing chamber and pass in front of the sources (18, 23), and in which process the plasma treatment occurs through charge carrier bombardment in a first step and the coating of workpiece (11) in a second step.

16. Process according to claim 14 or 15 in which the charge carriers consist of ions that are extracted from the arc discharge (18) directly with the aid of a negative workpiece voltage in such a way that they sputter etch the workpiece (11).

17. Process according to one of the claims 14 to 16 in which the homogeneity of the etch distribution across coating zone (b) can be set to predetermined values by selecting arc length, the distance (d) between the arc and the workpiece, the position of the arc relative to the workpiece, as well as by adjusting the plasma density distribution along the arc.

"Appendix A"

Fig. 5

Spindle torque [a.u]

End of tool life

High-voltage etching + 3.5 µm TiN (arc coating)

Low-voltage arc coating + 3.5 µm TiN (arc coating), (Invention)

Total milling depth [m]

Claims:

1. A tool with a tool body and a wear resistant layer system, said layer system comprising at least one layer of MeX, wherein

- Me comprises titanium and aluminum;
- X is at least one of nitrogen and of carbon

and wherein said layer has a Q_I value

$$Q_I \leq 2$$

and said tool is one of

- a solid carbide end mill;
- a solid carbide ball nose mill;
- a cemented carbide gear cutting tool, whereby the value of I(111) is at least 20 times the intensity average noise value, both measured according to MS.

2. The tool of claim 1 wherein there is valid for said Q_I:

$Q_I \leq 1$, preferably:

$Q_I \leq 0.5$, thereby preferably:

$Q_I \leq 0.2$, especially preferred:

$Q_I \leq 0.1$.

3. The tool of one of claims 1 or 2, wherein said MeX material is one of titanium aluminum nitride, titanium aluminum carbonitride, titanium aluminum boron nitride, thereby prefera-

bly one of titanium aluminum nitride and titanium aluminum carbonitride.

4. The tool of one of the claims 1 to 3, wherein Me further comprises at least one further element out of the group consisting of boron, zirconium, hafnium, yttrium, silicon, tungsten, chromium, thereby preferably of at least one of yttrium and silicon and boron.

5. The tool of claim 4, wherein said further element is contained in Me with a content i

$$0.05 \text{ at.\%} \le i \le 60 \text{ at.\%},$$

taken the content of Ti as 100 at.%.

6. The tool of one of the claims 1 to 5, further comprising a further layer of titanium nitride between said at least one layer and said tool body and wherein said further layer has a thickness d, for which there is valid

$$0.05\ \mu m \le d \le 5.0\ \mu m.$$

7. The tool of claim 6, wherein said layer system is formed by said at least one layer and said further layer.

8. The tool of one of the claims 1 to 7, wherein the stress within said at least one layer, σ, is

$$2 \text{ GPa} \le \sigma \le 8 \text{ GPa}, \text{ thereby preferably}$$

$$4 \text{ GPa} \le \sigma \le 6 \text{ GPa}.$$

9. The tool of one of the claims 1 to 8, wherein the content x of titanium in Me is:

70 at.% $\geq$ x $\geq$ 40 at.%, preferably

65 at.% $\geq$ x $\geq$ 55 at.%.

10. The tool of one of the claims 1 to 9, wherein the content y of aluminum in said Me is:

30 at.% $\leq$ y $\leq$ 60 at.%, thereby preferably

35 at.% $\leq$ y $\leq$ 45 at.%.

11. A method of producing a tool comprising a tool body and a wear resistant layer system, which comprises at least one hard material layer, comprising the steps of

- reactive PVD depositing said at least one layer in a vacuum chamber;

- selecting predetermined process parameter values for said PVD depositing beside of at least one of the two parameters consisting of partial pressure of a reactive gas in said vacuum chamber and of bias voltage of the tool body with respect to a predetermined reference potential;

- adjusting at least one of said partial pressure and of said bias voltage for realising said layer with a desired Q_I value and a value of at least one of the I(200) and I(111) to be at least 20 times larger than the average intensity noise value both measured according to MS.

12. The method of claim 11, further comprising the step of reducing said partial pressure for reducing said Q_I value and vice versa.

13. The method of claim 11 or 12, comprising the step of increasing said bias voltage for reducing said Q_I value and vice versa.

14. The method of one of the claims 11 to 13, further comprising the step of performing said reactive PVD deposition by reactive cathodic arc evaporation.

15. The method of claim 14, further comprising the step of magnetically controlling said arc evaporation.

16. The method of one of the claims 11 to 15, further comprising the step of depositing on said tool body a MeX layer, wherein Me comprises titanium and aluminum and X is at least one of nitrogen and of carbon and is introduced to said PVD depositing by reactive gas.

17. The method of one of claims 11 to 16, wherein said tool is one of

- a solid carbide end mill;

- a solid carbide ball nose mill;

- a cemented carbide gear cutting tool,

thereby selecting said Q_I value to be

$$Q_I \leq 2$$

by adjusting at least one of said reactive pressure and of said bias voltage for said reactive PVD depositing.

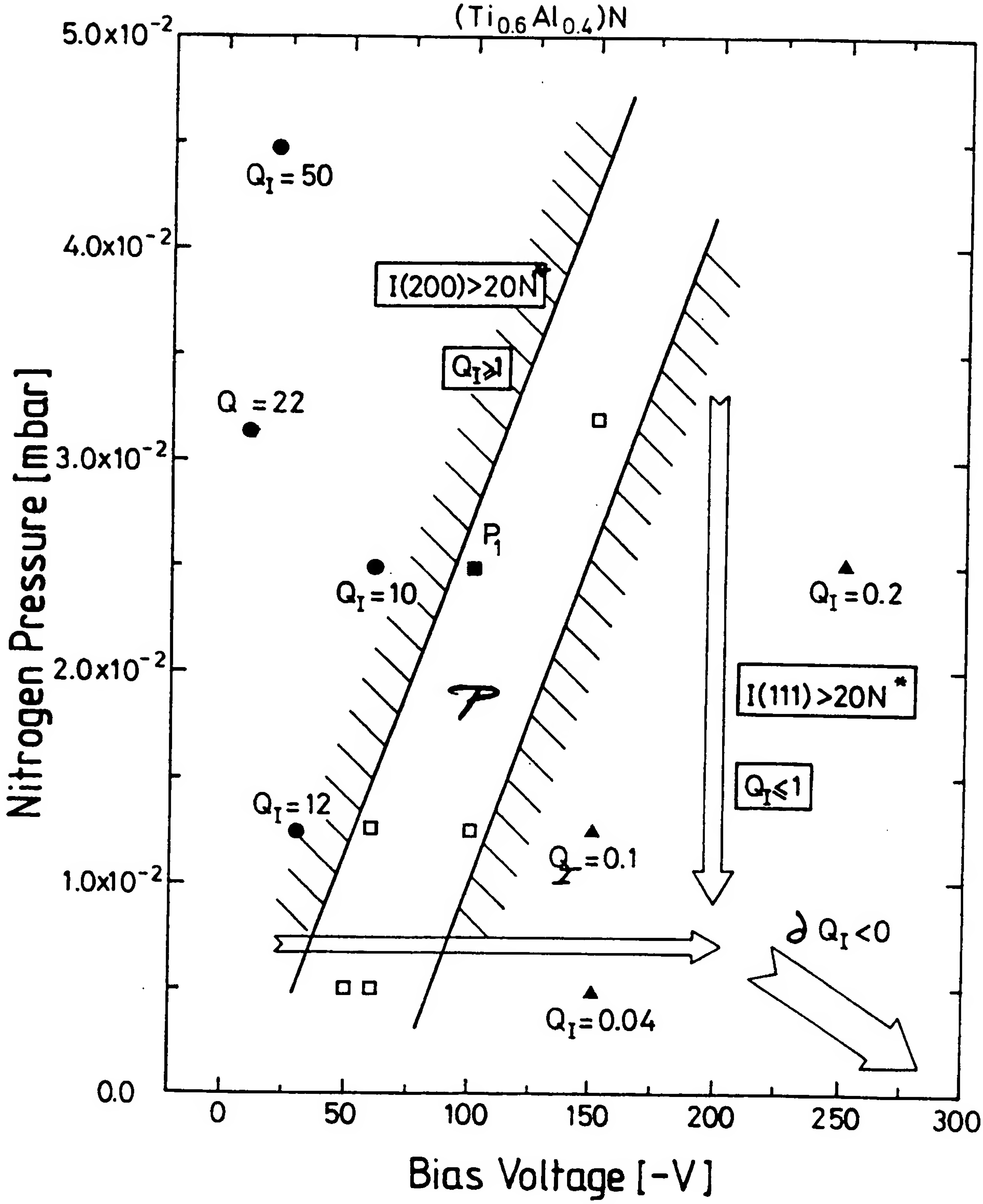
18. The method of claim 17, thereby selecting said Q_I value to be

$Q_I \leq 1$, preferably to be $Q_I \leq 0.5$ or even to be

$Q_I \leq 0.2$.

19. The method of claim 18, thereby selecting said Q_I value to be

$Q_I \leq 0.1$.



(Ti0.6Al0.4)N
5.0x10-2
4.0x10-2
3.0x10-2
2.0x10-2
1.0x10-2
0.0
Nitrogen Pressure [mbar]
QI = 50
Q = 22
QI = 10
QI = 12
P1
I(200)>20N*
QI≥1
I(111)>20N*
QI≤1
QI = 0.2
QI = 0.1
QI = 0.04
∂ QI < 0
0
50
100
150
200
250
300
Bias Voltage [-V]

FIG.1

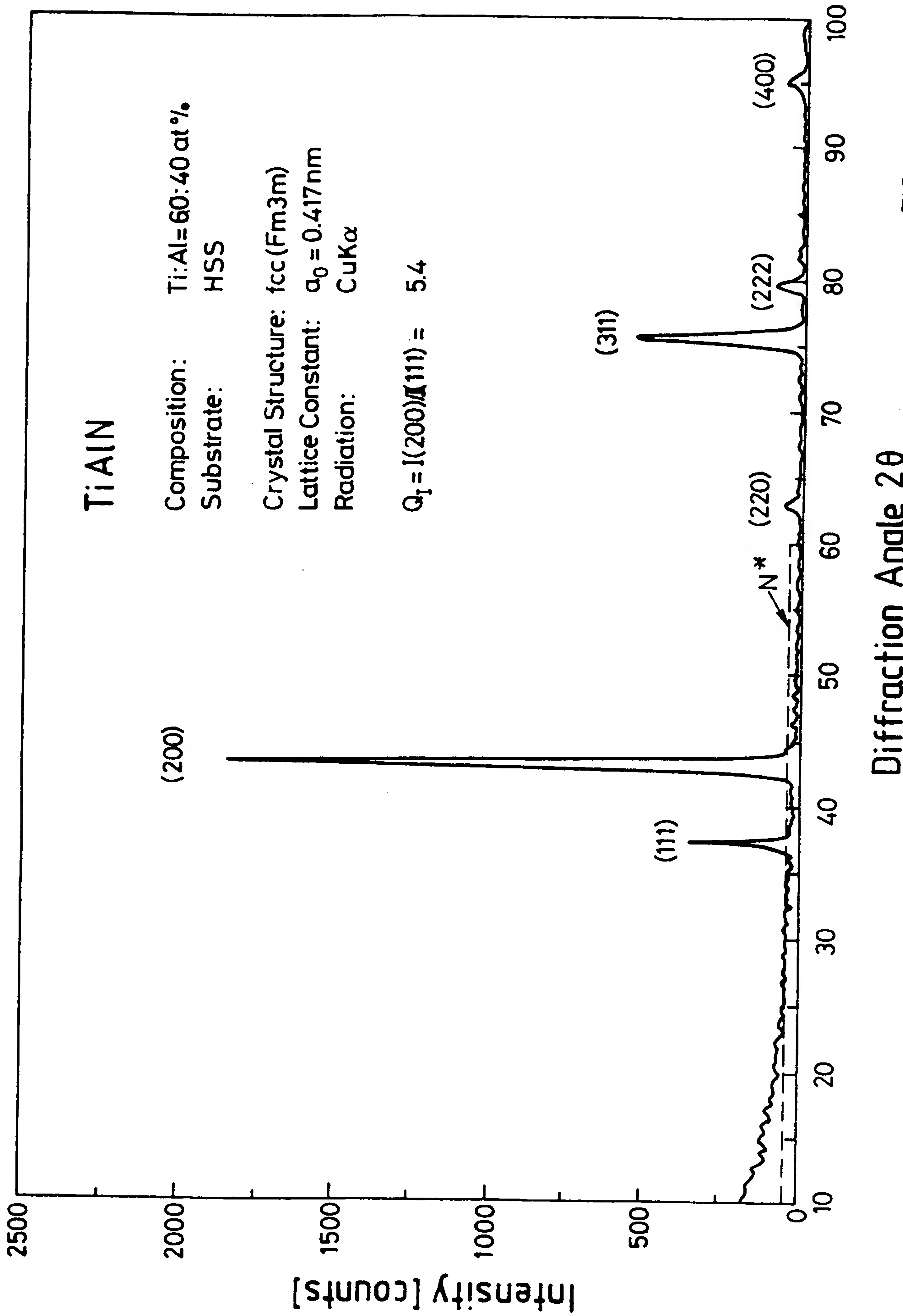

TiAlN
Composition: Ti:Al=60:40 at%
Substrate: HSS
Crystal Structure: fcc (Fm3m)
Lattice Constant: a0 = 0.417nm
Radiation: CuKα
QI = I(200)/I(111) = 5.4
(111)
(200)
N*
(220)
(311)
(222)
(400)
Intensity [counts]
2500
2000
1500
1000
500
0
10
20
30
40
50
60
70
80
90
100
Diffraction Angle 2θ

FIG. 2

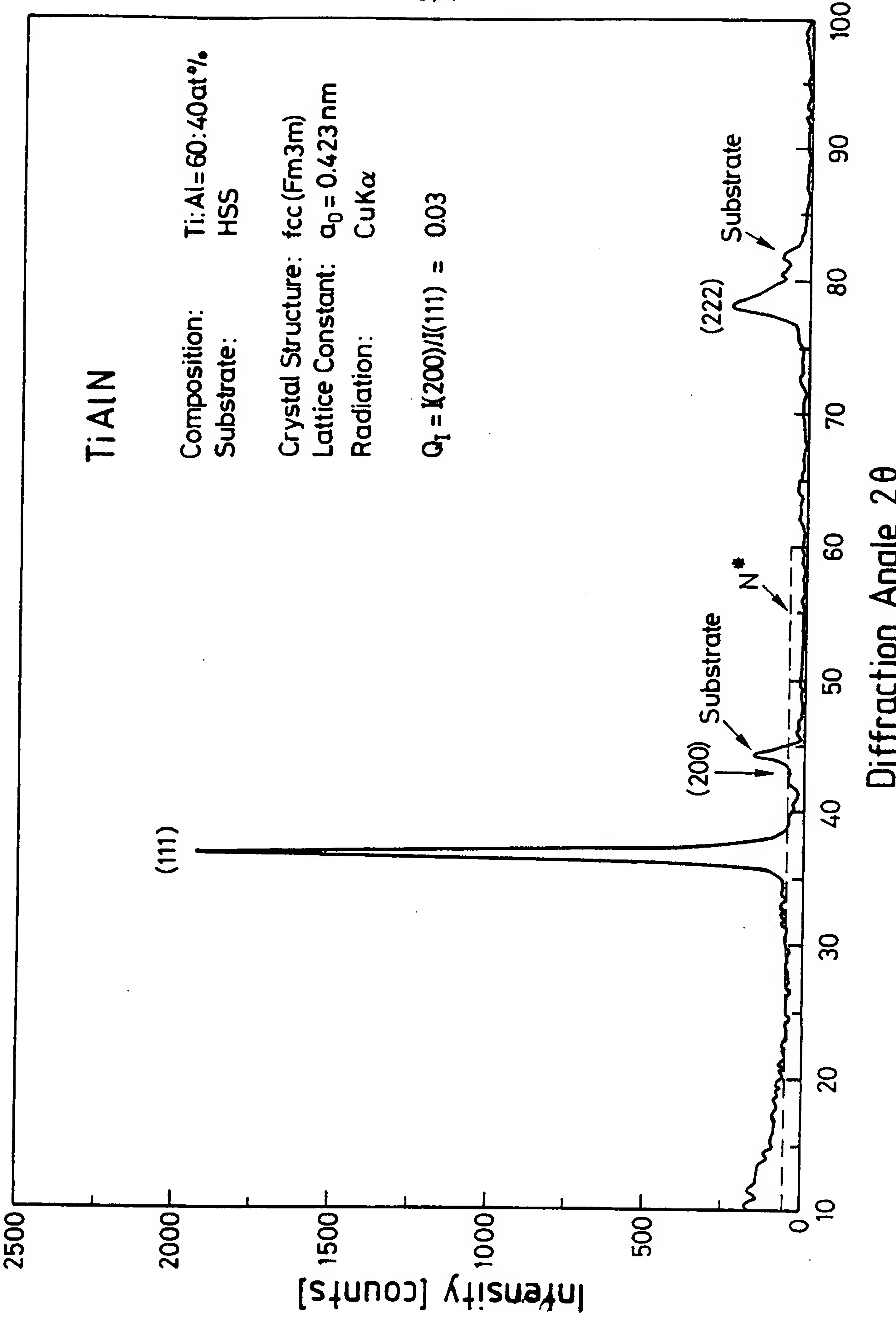

TiAlN
Composition: Ti:Al=60:40at%
Substrate: HSS
Crystal Structure: fcc (Fm3m)
Lattice Constant: a0 = 0.423 nm
Radiation: CuKα
QI = I(200)/I(111) = 0.03
(111)
(200)
Substrate
N*
(222)
Substrate
Intensity [counts]
2500
2000
1500
1000
500
0
10
20
30
40
50
60
70
80
90
100
Diffraction Angle 2θ

FIG.3

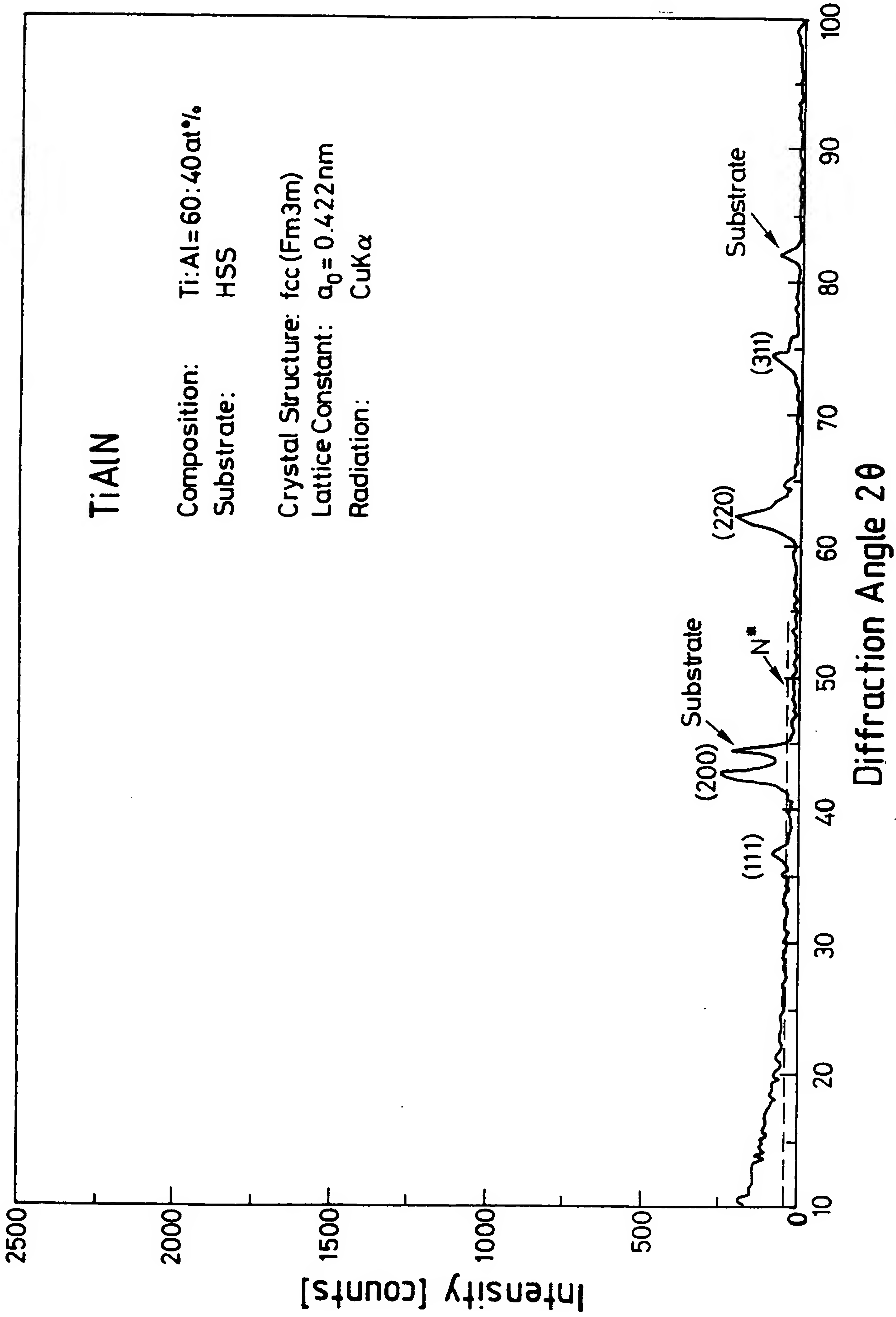

TiAlN
Composition: Ti:Al=60:40at%
Substrate: HSS
Crystal Structure: fcc (Fm3m)
Lattice Constant: a_0 = 0.422nm
Radiation: CuKα
(111)
(200)
Substrate
N*
(220)
(311)
Substrate
Intensity [counts]
2500
2000
1500
1000
500
0
10
20
30
40
50
60
70
80
90
100
Diffraction Angle 2θ

FIG. 4

"Appendix A" 1/4

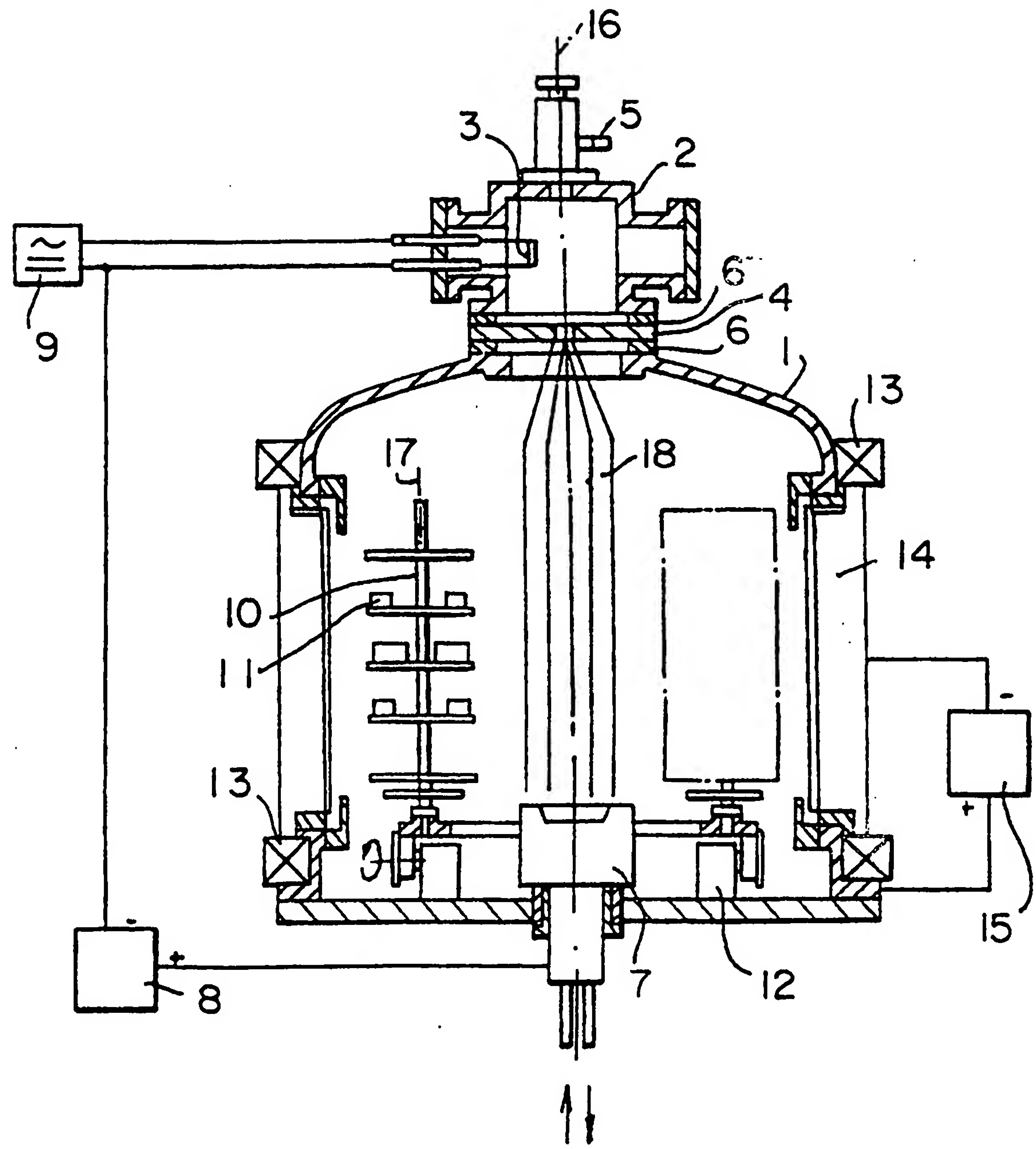


FIG. 1

"Appendix A"

FIG.2

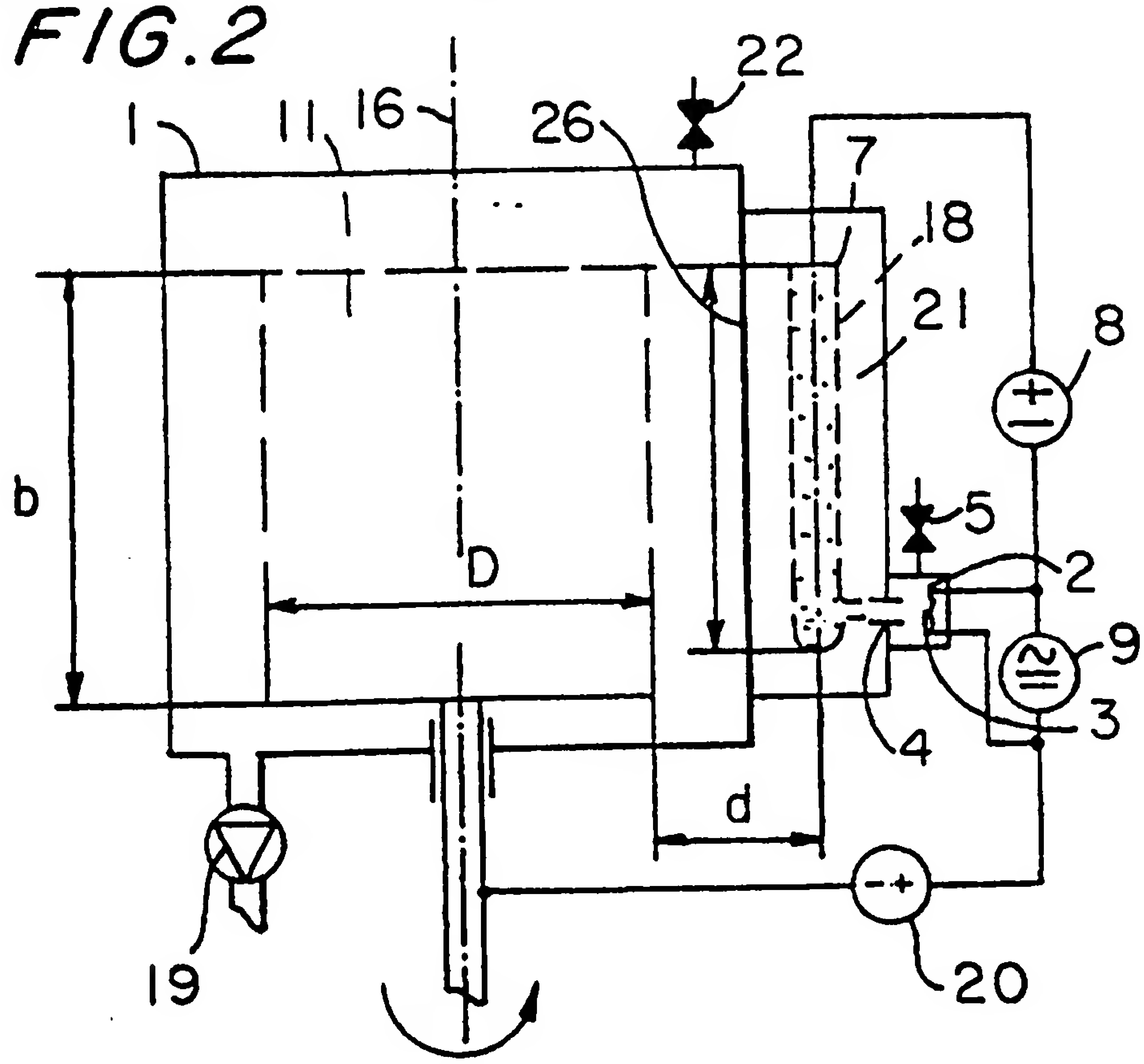
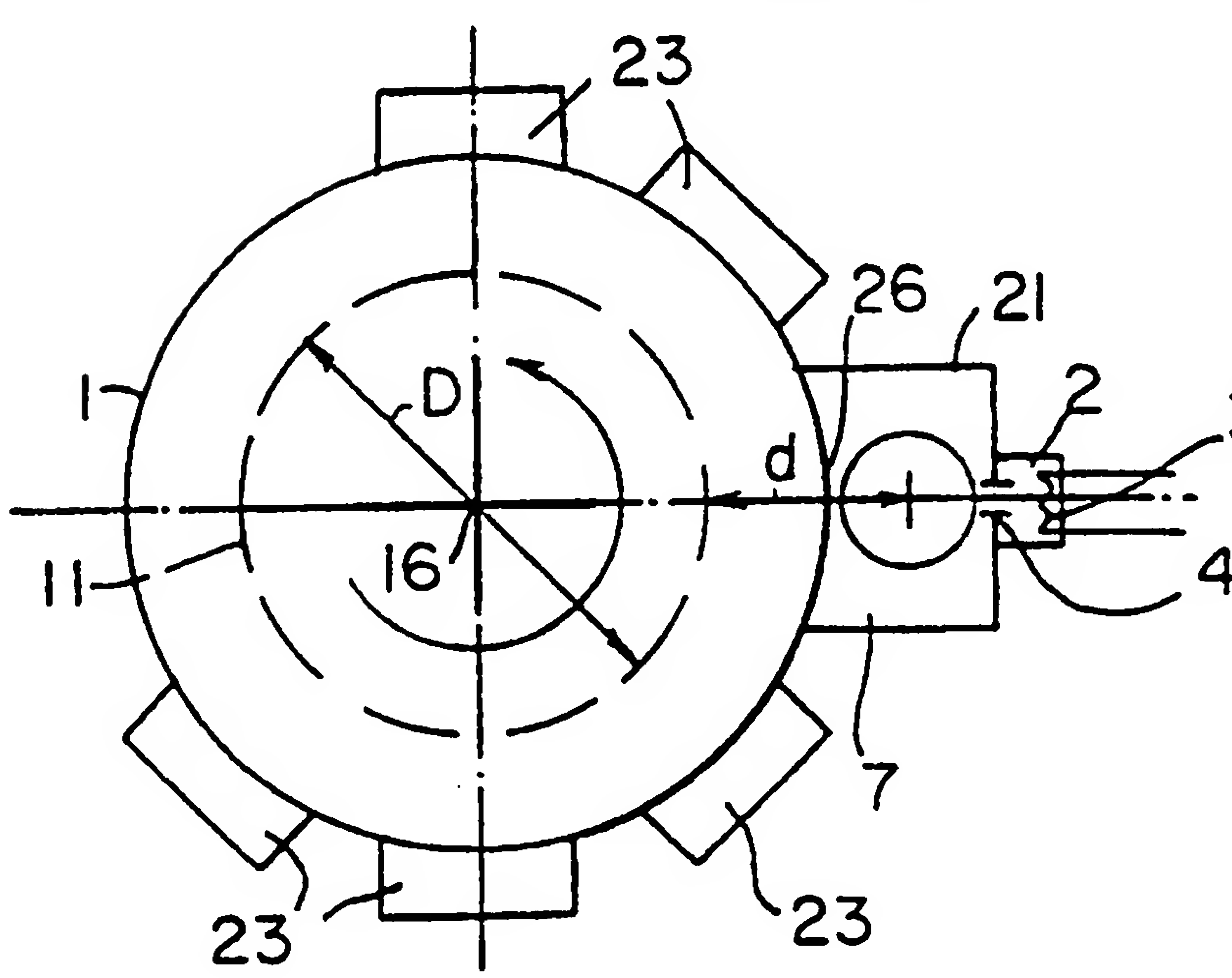


FIG.3



"Appendix A" 3/4

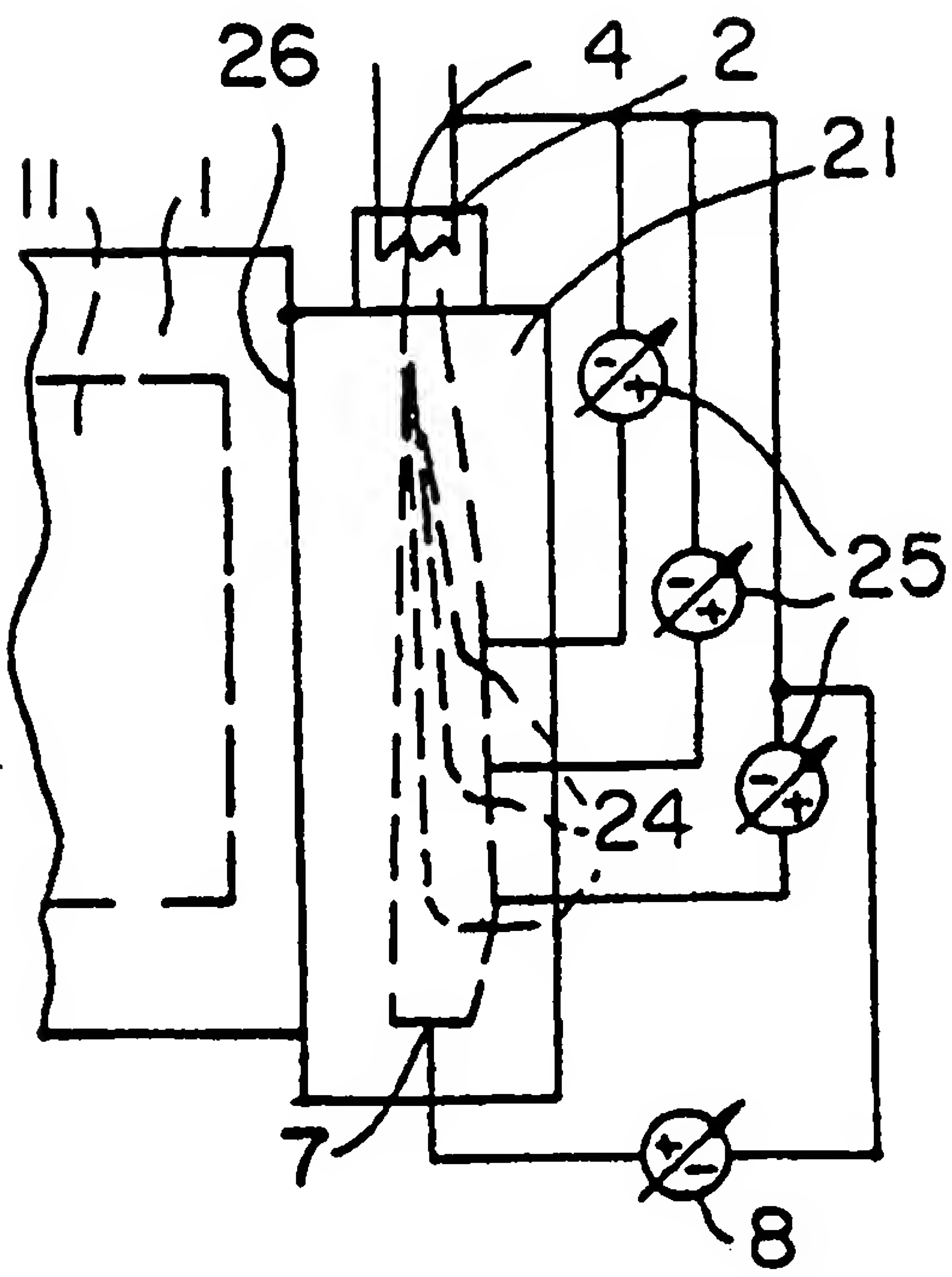


FIG. 4a

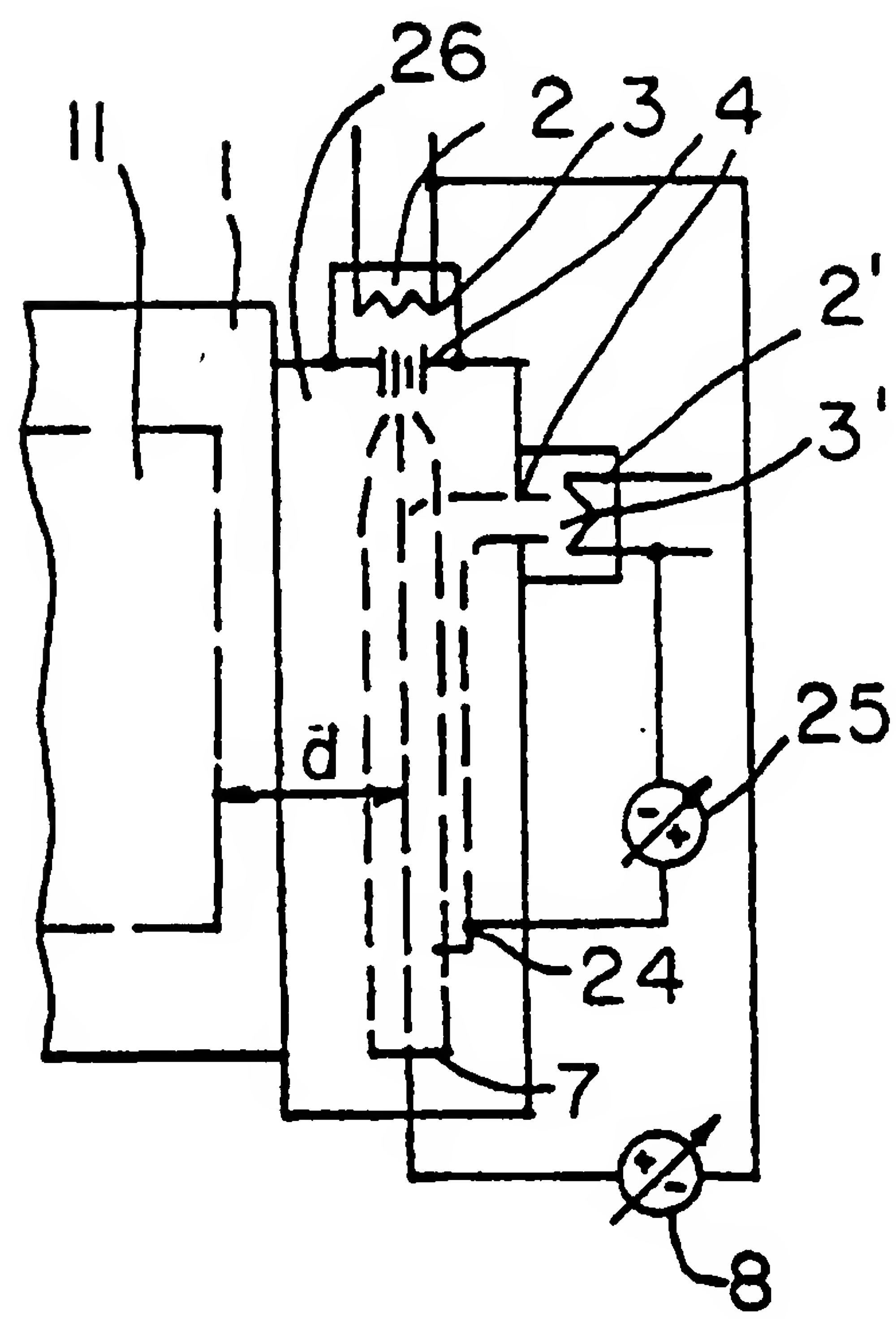


FIG. 4b

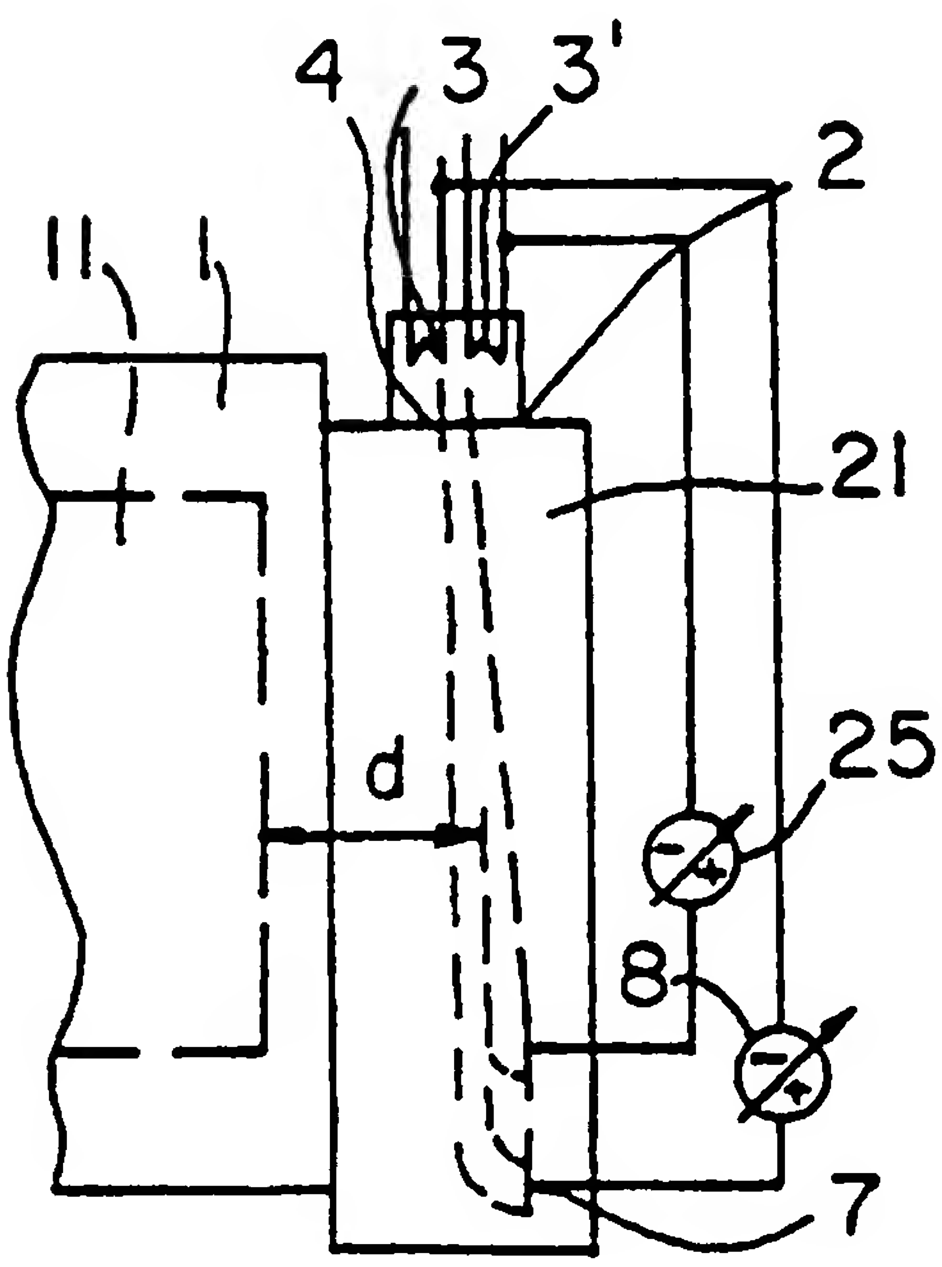
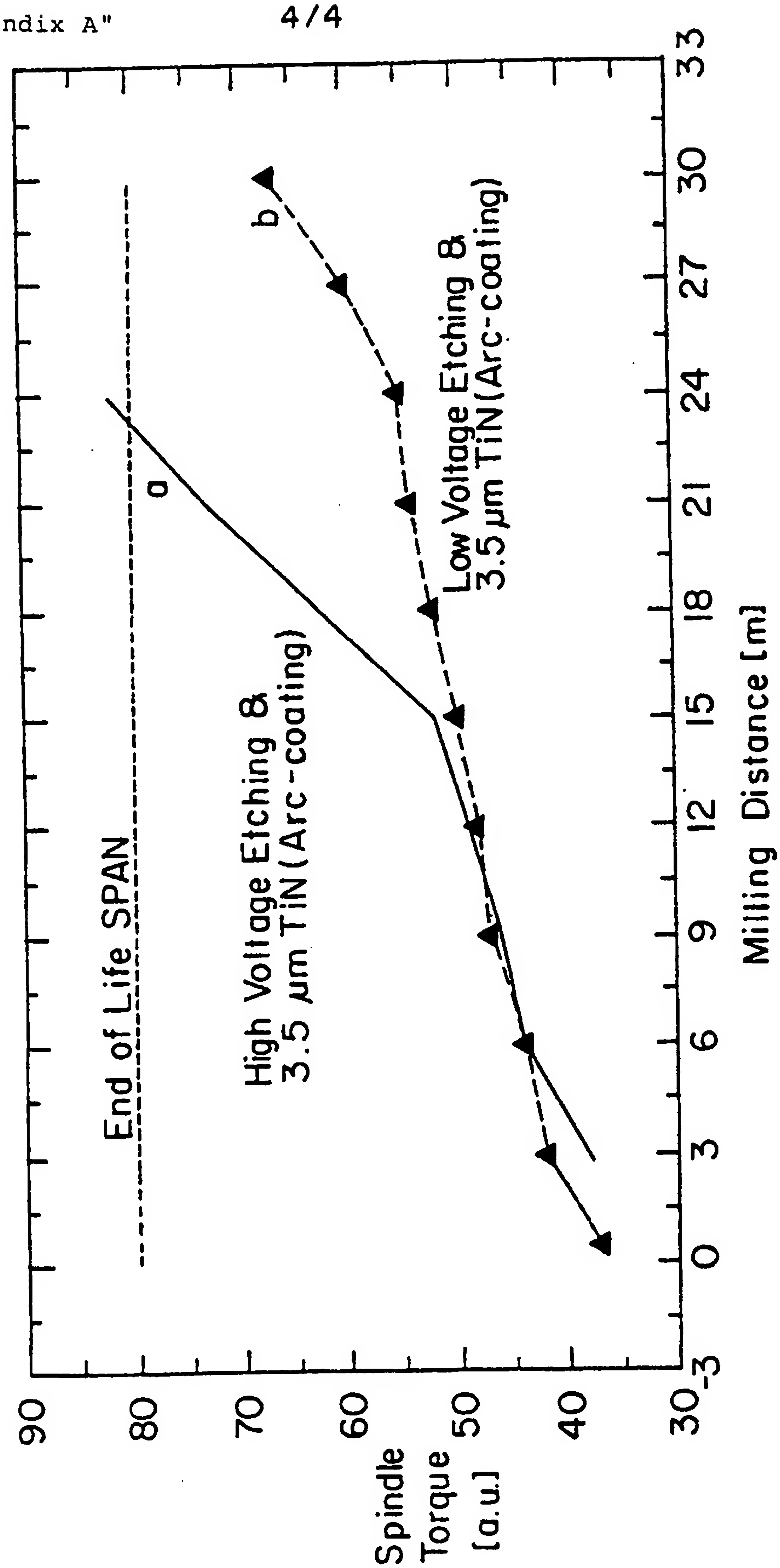


FIG. 4c

"Appendix A" 4/4

FIG. 5



INTERNATIONAL SEARCH REPORT

International Application No
PCT/IB 97/01090

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C23C14/06 C23C14/00 C23C14/54 B23C5/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PETROV I ET AL: "AVERAGE ENERGY DEPOSITED PER ATOM: A UNIVERSAL PARAMETER FOR DESCRIBING ION-ASSISTED FILM GROWTH?" APPLIED PHYSICS LETTERS, vol. 63, no. 1, 5 July 1993, pages 36-38, XP000382556	11,16
Y	see the whole document	12-15, 17-19
X	PATENT ABSTRACTS OF JAPAN vol. 096, no. 012, 26 December 1996 & JP 08 209335 A (HITACHI TOOL ENG LTD), 13 August 1996, see abstract	11,16
Y	EP 0 591 122 A (SANDVIK AB) 6 April 1994 see page 2, line 40 - line 52	1-3,9, 10,17-19

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
14 May 1998	22/05/1998

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Ekhult, H

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
Y	ADIBI F ET AL: "EFFECTS OF HIGH-FLUX LOW-ENERGY (20-100 EV) ION IRRADIATION DURING DEPOSITION ON THE MICROSTRUCTURE AND PREFERRED ORIENTATION OF TI0.5AL0.5N ALLOYS GROWN BY ULTRA-HIGH-VACUUM REACTIVE MAGNETRON SPUTTERING" JOURNAL OF APPLIED PHYSICS, vol. 73, no. 12, 15 June 1993, pages 8580-8589, XP000381378 see paragraph B-D ---	1-3,9,10
A	KNOTEK O ET AL: "THE STRUCTURE AND COMPOSITION OF TI-ZR-N, TI-AI-ZR-N AND TI-AI-V-N COATINGS" MATERIALS SCIENCE AND ENGINEERING A: STRUCTURAL MATERIALS: PROPERTIES, MICROSTRUCTURE & PROCESSING, vol. A105/106, 1 January 1988, pages 481-488, XP000108123 see paragraph 5; figure 8 ---	4,5
A	EP 0 701 982 A (SUMITOMO ELECTRIC INDUSTRIES) 20 March 1996 see column 14, line 6 - line 11 ---	6,7
Y	ROOS J R ET AL: "INTERRELATIONSHIP BETWEEN PROCESSING, COATING PROPERTIES AND FUNCTIONAL PROPERTIES OF STEERED ARC PHYSICALLY VAPOUR DEPOSITED (TI,AL)N AND (TI,NB)N COATINGS" JOURNAL OF THE LESS-COMMON METALS, vol. 93 / 194, no. 1 / 02, 1 December 1990, pages 547-556, XP000168996	14,15
A	see page 553, line 4 - line 16 ---	8
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/IB 97/01090

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